# The Journal of the

# SOCIETY OF DYERS AND COLOURISTS

Volume 73 Number 4

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Manuscripts submitted for publication in the Journal should be typewritten (double spacing) on good-quality paper, using one side of the paper only and leaving a margin at least I in. wide on the left-hand side. The time taken in refereeing papers (both lectures and communications) will be reduced to a minimum when authors submit two copies of the typescript.

in view of the high costs of publication, it is essential that authors should be as concise as possible. When experimental procedure has already been published, a literature reference to the paper containing the details is sufficient, whilst well known experimental methods should be described very briefly.

introductory paragraphs describing the aims of the investigation and the method of attack are desirable, and should be followed by the experimental results and their discussion. There should be a brief summary for insertion at the beginning of the paper. References to the literature should be numbered consecutively, using superscript numbers without brackets immediately following the text words or author's name to which they refer.

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The number of figures and graphs should be kept as low as possible, and data should be presented in the form of either tables or graphs, not both. Drawings should be carefully prepared, preferably in Indian Ink, on plain white drawing paper er, preferably, Bristol board. In graphs, the frame and actual curves should be ruled and Inked more heavily than any co-ordinate lines, and the latter should not be close together as in ordinary graph paper. Experimental points should always be given, and where several graphs appear in a single Figure clear means of differentiation must be adopted. All numbers and legends are set up in type by the printer, and authors should therefore indicate them lightly in pencil.

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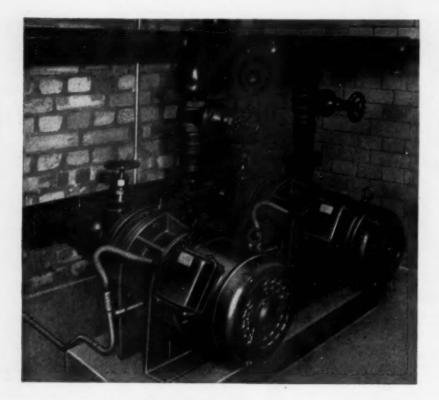
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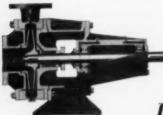
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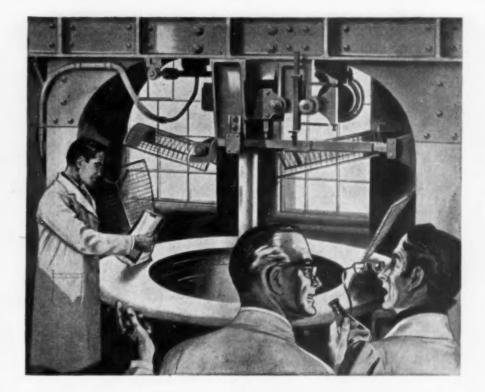


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### Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal* —

### LECTURES

- Fibro and Fibrolane in the Carpet Trade with particular reference to Dyeing R. C. Cheetham
- A New Approach to the Continuous Dyeing of Secondary Cellulose Acetate,
  Cellulose Triacetate, and Terylene Polyester Fibre the Vapocol Process

  D. A. Garrett
- The Dyeing of Dicel and Tricel Yarns and Fabrics
- H. C. Olpin and J. Wood

Dyeing (George Douglas Lecture)

G. S. J. White

### COMMUNICATIONS

- The Dyeing of Cellulose Acetate with Disperse Dyes-VI
- C. L. Bird and Miss P. Harris
- Determination of the Migratory Properties of Direct Dyes

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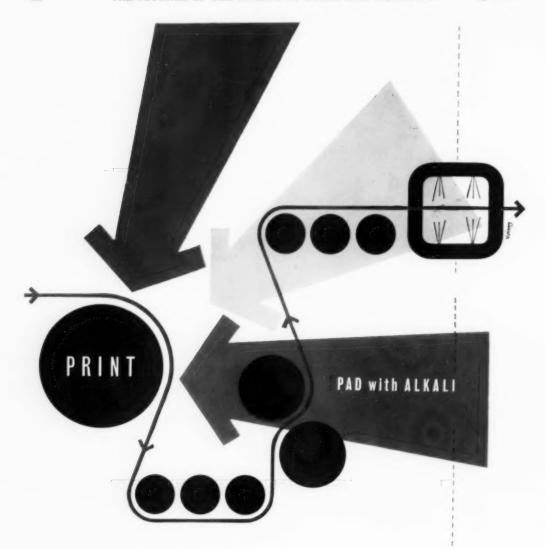
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Volume 73 Number 4

**APRIL 1957** 

Issued Monthly

PUBLICATIONS SPONSORED BY THE SOCIETY'S FASTNESS TESTS CO-ORDINATING COMMITTEE—XXII\*

### A Critical Appraisal of the International Daylight Fastness Test

The international daylight fastness test has been critically examined to determine its reproducibility and its reliability. Only one variable factor in exposure—the effective humidity—has been found to cause variations in assessment, and this is governed primarily by the proportion of sunlight falling on the pattern during exposure. This causes the assessments of humidity-agnitive materials to be up to 2 grades higher in sunny countries than in predominantly cloudy regions. This variation can be eliminated only at the expense of the reliability of the result, a solution which cannot be accepted. There is evidence to suggest, however, that the reproducibility of the method would be very good for the majority of fabrics, whose sensitivity to humidity, like that of the standards, is low. The test is a reliable guide to the expected resistance to fading which will occur during use, even of sensitive materials, providing that the daylight test is carried out in the same country as that in which the fabric is to be used or in one whose climate is similar and providing that the fabric does not become wetted during use.

### Introduction

During the past eight years there has been much international discussion of the determination of the light fastness of coloured textiles, and now a method has been formally accepted by the International Organisation for Standardisation (I.S.O.) which should eventually be adopted by most of the thirty-seven countries which are members of I.S.O. In the United Kingdom the test has already been adopted as B.S. 1006: 1955, which is fundamentally similar to the method first published in 1952<sup>1</sup>; it will not be adopted by the U.S.A., however, as it would render invalid all the existing data obtained by using the AATCC method and standards <sup>2</sup>.

There is no doubt that light-fastness testing is one of the most important of all fastness tests, and it is also one which must by its very nature be fundamentally different in conception and execution from the others, which are themselves fundamentally similar. Because of these considerations, therefore, the author felt that the test should be subjected to a detailed critical examination, as it was hoped that the I.S.O. Recommendation would not require any major alterations for many years.

The two fundamental characteristics which a fastness test should possess are reproducibility and reliability, and these two features were therefore studied.

### I— Reproducibility of the Daylight-exposure Method

In theory, the simplest method of investigating the reproducibility of the test would be to select a large number of patterns at random, expose them under several widely different conditions, and analyse the results; however, this method has never appealed to the author, for several reasons—

- (1) The extremely large number of dye-fibre– aftertreatment combinations commercially available, so that a sample which is statistically satisfactory would probably be unmanageable
- (2) The difficulty of appraising the results: for example, if only 1% of the patterns gave results which varied by more than half a grade, this might be considered satisfactory on statistical grounds; but if, for example, all crease-resisted direct-dyed spun rayon-staple fabrics were in this minority, then producers of such materials would take an entirely opposite, but equally justified, view
- (3) The difficulty of ensuring that all the possible combinations of conditions have been covered.

In the past, no other method of appraisal could be used, but during the last few years sufficient has been learnt about the possible causes of anomalous results in light-fastness testing to make it feasible to investigate the reproducibility of the method by a different and much more attractive approach: this is to consider first which variables in daylight testing could cause anomalies, to select patterns known to be highly sensitive to each of these, and then to expose them under various conditions such that the effect of each variable is in one case as great as possible and in another as low as possible. By analysing the results a much more satisfactory appraisal of the test can be made, and

Part of the information given in this paper and in XVII and XXI has formed the basis of lectures to the Manchester, West Riding (in Leeds), and Northern Ireland Sections.

This paper was submitted for consideration at the fourth meeting of I.S.O. Subcommittee ISO/TC 38/SC 1 haid in New York in September 1956 (Document 134).

experiments with this object in view were started in November 1953.

### CAUSES OF ANOMALOUS RESULTS IN DAYLIGHT

A piece of coloured material, together with the standards, is exposed to daylight in accordance with the official method either in two different places, at two different times, or both: in one case, the pattern is reported as having a light fastness of 4, in the other 6. What are the possible reasons for this anomaly and which is the correct result?

Firstly, there is the possibility of errors in assessment; i.e. two people might report different results even if they assessed the same exposure card. Similarly, the possibility of one person assessing the same card differently on two different occasions must also be borne in mind. A statistical study of errors of this kind would be very well worthwhile and undoubtedly revealing, but was considered to be outside the scope of the present investigation.

Secondly, there is always the possibility of contamination during one exposure, e.g. by sulphur dioxide in industrial districts.

However, there are anomalies which cannot be explained by either of these factors and which are therefore true photochemical anomalies; these must be due to variations in one or more of the following factors—

- (i) The spectral composition of the light
- (ii) The temperature during exposure
- (iii) The moisture content during exposure.

Each of these possibilities has been studied in connection with the anomalies between fading lamps and daylight <sup>3, 4</sup>, and the first stage in the present investigation was to consider to what extent they could cause anomalies in daylight testing.

### (i) Spectral Composition of the Light

The intensity of radiation falling on an exposed surface depends upon a wide variety of factors, such as latitude, altitude, time of day, season of year, as well as local atmospheric conditions of cloudiness, etc. The range of intensities is obviously extremely great, but, although there is some evidence bethat fading is not always proportional to the intensity of the radiation, this has never been shown to be a cause of anomalies in testing and, in the opinion of the author, it is unlikely to be so.

Anomalies would be expected, however, if the balance of actinic radiation varies, e.g. if the light falling on a pattern in one exposure is much richer in ultraviolet radiation than in another; this has already been shown to be a contributory cause of anomalies when a carbon arc lamp is used <sup>3</sup>. Such variations are possible in daylight testing, because the climatic and geographical factors which reduce the total intensity by absorption and scattering do not affect all wavelengths equally, the effect being greatest on the shorter wavelengths. For example, Forsythe and Christison <sup>6</sup> compared clear days in

Ohio (U.S.A.) in December and in June: whilst the increased absorption in winter reduced the total solar radiation to about one-third, it reduced the intensity of radiation below  $325 \,\mathrm{m}\mu$ , to one-twentieth, and that below  $310 \,\mathrm{m}\mu$  to less than one-hundredth, of the summer value.

However, these variations are not likely to be a cause of anomalies in daylight testing, for the radiation which is absorbed to a significantly greater extent than average lies below 330 m $\mu$ . and the amount reaching the pattern even under the best conditions is minute, partly because the amount present in solar radiation falling on the earth's surface is very small (that below 325 m $\mu$ . amounting to only 0.25% of the total solar radiation even under the clearest conditions), but mainly because window-glass absorbs this radiation very strongly, viz. about 80% at 320 m $\mu$ . and about 98% at 310 m $\mu$ .

It is commonly believed that the amount of ultraviolet radiation present in daylight in winter or during cloudy weather is negligible, but this is true only of the extreme short-wave ultraviolet occurring in daylight; the bulk of the ultraviolet, viz. that of 330–380 m $\mu$ ., is not reduced more than visible light.

The widespread belief that there is little ultraviolet radiation in winter probably arises from the fact that browning of the human skin is extremely slow in winter in the United Kingdom. However, although browning is loosely attributed to "the ultraviolet", it is in fact due entirely to the shortwave ultraviolet, wavelengths greater than 315 m $\mu$ . having a negligible effect, and the greatest efficiency being at 295 m $\mu$ , the limit of solar radiation at the earth's surface?

### (ii) Temperature during Exposure

It has already been shown that differences in temperature are unlikely to be a direct cause of anomalies in testing, although complete exoneration of this possibility awaits the outcome of an investigation which is being carried out in the U.S.A. Indirectly, however, temperature is of extreme importance, as it is the chief factor determining the moisture content during exposure.

### (iii) Moisture Content during Exposure

This factor was found to be potentially the most important cause of gross anomalies between daylight and fading-lamp exposures, and variations of as much as 4 grades could be directly attributed to differences in the moisture content of the fabric during exposure 4. The variations arise because, although the rate of fading of the light-fastness standards is independent of their moisture content during exposure, this is not true for many dye-fibre combinations, which fade much faster when the surface moisture content is high than when it is low.

Thus the only factor which is likely to cause significant variations in daylight testing is the moisture content during exposure. The reproducibility of the method can therefore be ascertained by selecting a pattern whose rate of fading increases regularly and rapidly with increasing moisture

content, and exposing it against the standards under variable conditions chosen to ensure that the moisture content is in one case the minimum and in another the maximum possible. The test specifies certain conditions of exposure, e.g. facing south, under glass at 45°, but does not specify when or where; it is by varying the time and the place that the moisture content must be varied.

### EXPERIMENTAL

It is obvious that the discovery of the pattern of maximum sensitivity would require the exposure at high aud low humidities of literally thousands of patterns, which would have been impracticable. However, in 1953 a case of anomalous fading was investigated which showed that the dye Brentamine Fast Scarlet R→Brenthol AN (ICI) had very high sensitivity. A medium depth of this azoic combination on mercerised poplin was therefore used for all the initial tests.

In some cases, dyeings of basic and vat dyes of high sensitivity were also included, but their

TABLE I Time of Exposure Place Light Months Fastness Year UNITED KINGDOM 1953-1954 Nov.-April Blackley, Manchester 4 Wilmslow, Cheshire Wilmslow, Cheshire Leeds, Yorkshire April-June 4-5 1954 4.5 July July-Aug. 4-5 1955 May-June Wilmslow, Cheshire 5 Blackley, Manchester 5 June-Aug. Wilmslow, Cheshire 5 5 Glasgow Manchester City 5 Didsbury, Manchester 5 Wembley, Middlesex 75 Clayton, Manchester 5 Wilmslow, Cheshire Blackley, Manchester Blackley, Manchester July-Aug. 6 July-Sept. Sept.-Mar. 4-5 4-5 1956 Blackley, Manchester Jan.-May May Glasgow Loods 5 Wilmslow, Cheshire 5 Wembley, Middlesex OVERSEAS 1954-1955 Nov.-Feb. Bombay, India 6-7 1955 June-Oct. Providence, R.I., U.S.A. 6 1955-1956 Dec.-Mar. Providence, R.I., U.S.A. 6 1956 Feb.-April Bombay, India 6-7 May May-July Bergen, Norway 4.5 Melbourne, Australia 4-5 May-June Gothenburg, Sweden Leverkusen, Germany May-Aug. Copenhagen, Denmark Providence, R.L., U.S.A. Delft, Holland 5 6 May-June May 5-6 6 May-June Verviers, Belgium 6 Basle, Switzerland May-July 65 May-June Toronto, Canada May-June Villers-St. Paul (Oise), 43 France May-Aug. Johannesburg, 63. Africa May Cairo, Egypt 6 - 7May-June Milan, Italy 6-7 May-June Aigues-Mortes (Gard), 6 - 7France St. Clair (Isere), France 6-7

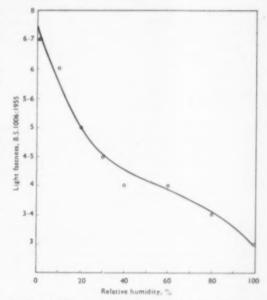


Fig. 1-Light Fastness of Sensitive Pattern at Different R.H. Values

behaviour was found to be similar to that of the red azoic combination. In the interests of simplicity, therefore, only the results obtained with the azoic combination are given here.

The sensitivity of the azoic combination was determined by exposing it to daylight together with the standards in sealed bottles containing air at controlled relative humidities: the results are shown in Fig. 1. This pattern could therefore reveal variations of as much as 3½ grades, and in fact such variations were found 3 between various fading lamps.

The results obtained by exposing this pattern against the standards by the internationally accepted method in various parts of the world at various times are given in Table I.

#### DISCUSSION OF RESULTS

The results in Table I show that variations of 2½ grades (from 4 to 6–7) are possible in daylight testing by the internationally accepted method. Such large variations are undoubtedly of considerable importance and must be carefully considered, but it must be emphasised immediately that they will occur only with patterns of high sensitivity; the majority of dye-fibre combinations are of much lower sensitivity, and in such cases the maximum variations will be much less; where the sensitivity is as low as that of the standards, identical results should be obtained at all times and in all places.

The wide variations in the results are caused by the differences in the surface moisture content during exposure. It was shown previously 4 that this is governed, not by the relative humidity of the air surrounding the pattern, as one might expect, but by the effective humidity, a complicated factor which is almost entirely dependent on the ratio of bright sunshine to total daylight

July-Aug.

Bombay, India

TABLE II

	Month	Average l	R.H. (%)	Average f	Sunshine (day)	
Manches	ter Bombay	Manchester 9.0 a.m.	Bombay 8-30 a.m.	Manchester	Bombay	
April	November	73	73	3.8	9-3	
May	December	71	70	4.6	9-7	
June	January	71	70	5.2	9-8	
July	February	76	71	4-4	10.2	
August	March	77	73	3.7	10.0	
		TABLE	: 111			
	35	A	D TF (0/)	A		

7	Ionth	Average 1	R.H. (%)	Average Sunshine (hr./day)		
Manchester	Bombay	Manchester 9-0 a.m.	Bombay 8-30 a.m.	Manchester	Bombay	
December	December July		83	0.2	2-3	
January	August	85	83	0-4	3.2	
February	September	83	85	1.9	5-5	

falling on the pattern during exposure: the greater the proportion of sunshine, the lower the effective humidity, and vice versa. The results in Table I are in general agreement with this conclusion, the highest values being obtained in the sunniest regions.

More detailed confirmation is afforded by the exposures made in Blackley and in Bombay, for summer exposures in Blackley gave values of 5, whereas winter exposures in Bombay gave 6-7, which shows that the effective humidity in India is much lower. The relative humidities in the two places are, however, similar, but Bombay is much sunnier, as shown in Table II. The sunshine figures were obtained from Brooks s, and the relative humidity figures from data in the possession of the Meteorological Office (Air Ministry), London.

TABLE IV

	TANKS AV	
Country	Town	Bright Sunshine (hr./day)
Great Britain	Manchester	2.9
	London	3.7
U.S.S.R.	Moscow	1.9
Denmark	Copenhagen	4-0
Holland	Utrecht	4-3
Belgium	Brussels	4-4
Germany	Berlin	4-4
Switzerland	Basle	4.6
Austria	Vienna	4-9
France	Paris	4.9
Sweden	Stockholm	4.9
China	Hong Kong	5-4
Canada	Ottawa	5-5
New Zealand	Wellington	5-7
Japan	Tokio	5-8
Australia	Melbourne	6-1
Italy	Rome	6-4
Argentina ,	Buenos Aires	6-6
U.S.A.	New York	7-3
Portugal	Lisbon	7.5
South Africa	Cape Town	7-5
Greece	Athens	7-6
India	Bombay	7-9
Spain	Madrid	8-0
U.S.A.	San Francisco	8-0
Egypt	Cairo	10.3
The state of the s		

Perhaps the most convincing confirmation of all is the observation that a higher light-fastness assessment (5) was obtained from an exposure made in Bombay during July and August than from one made in North-west England during the winter (4 and 4-5). This means that the effective humidity in North-west England in winter is actually higher than that in Bombay during the monsoon period; the reason is again found in the sunshine figures, as shown in Table III.

Although several of the results recorded in Table I indicated that the effective humidity during exposure was the minimum possible (approx. 0%), the maximum was never reached, as Fig. 1 shows that at 100% effective humidity the light fastness of the sensitive pattern would be 3. It seems fairly safe to assume that the highest effective humidity will be found in industrial areas of North-west England during the winter months, not because the average relative humidity is higher than elsewhere (it is actually lower than in most coastal districts of the United Kingdom), but because the amount of sunshine is smaller.

### IMPLICATIONS OF LOW REPRODUCIBILITY

It has been shown that the low reproducibility of the test occurs with patterns which are sensitive to differences in moisture content on exposure. This is due to the differences in the ratio of sunshine to total daylight which occur in various parts of the world, and sunshine figures for several important towns are given in Table IV, the data being obtained from Brooks 8, the Meteorological Office 9 and Uttinger 10. The greatest variation observed was 21 grades (from 4 to 6-7), but because Standard 7 is considerably more than twice as fast as Standard 6, variations of 3 grades must be expected with other equally sensitive patterns, the highest results being obtained when the ratio of sunshine to total daylight is high (e.g. Cairo, 5:6) and the lowest when it is low (e.g. Manchester, 1:8).

One method of improving the reproducibility is immediately apparent: this is to employ the exposure method described in the AATCC Sunlight Test <sup>11</sup>, and this possibility must obviously be very carefully considered.

The American test is based on exposure against standards (which, incidentally, are quite different from the I.S.O. standards), but the time of exposure is restricted: thus, whilst the I.S.O. method is to expose continuously day and night at any time of the year, the AATCC method restricts exposure to "the hours of 9 a.m. and 3 p.m. (Standard Time) on sunny days between 1st April and 1st October". The reason for this restriction is that "These standard conditions have been found to give the best duplicability of results with respect to the amount and character of fading in sun tests" this has been amplified by Appel and Smith, who state that it was designed to avoid "the low intensity radiation and high atmospheric humidity occurring at intervals during continuous exposure".

It is obvious that the claim for greater reproducibility of the restricted method must be conceded. In fact, during the exceptionally sunny summer of 1955 it was practicable to make exposures by this method in Blackley, and the sensitive pattern was found to have the same light fastness as when tested by this method in Providence, Rhode Island, U.S.A., viz. 6–7.

However, this cannot be accepted as a satisfactory solution, as, not only would it be impracticable in the United Kingdom, where the number of days meriting the description "sunny" are usually too few, but the method would achieve a high degree of reproducibility only at the expense of reliability, as the effective humidity would be very much lower than would occur during continuous exposure: even this is somewhat lower than occurs when fabrics are exposed in daily use (see below); test conditions which give a result of 6–7 when the maximum under conditions of use is only 5 would obviously not be tolerated in the United Kingdom and other predominantly cloudy countries.

Within one country, the reproducibility is naturally very much better, as the variations in the ratio of bright sunshine to total daylight will be much less. Thus in the United Kingdom, although the maximum variation was 2 grades, the two extremes were from exposures made under exceptional conditions: the result of 4 was obtained from a winter exposure, and these are rarely made, because, not only is the fading rate very low, but in populous districts soiling and atmospheric degradation are serious problems; the exceptionally high result of 6 was obtained during an exceptionally sunny period, July 1955 being the sunniest July on record in the North-west of England <sup>12</sup>.

Taking into account, therefore, the paramount importance of reliability, the fair degree of reproducibility even with sensitive fabrics within one country, and the fact that the average reproducibility of all dyed and printed fabrics will be much greater leads to the conclusion that the test should not be modified.

### II— Reliability of the Daylight Testing Method

The criterion of reliability in the case of tests such as carbonising or washing is simple: does the test produce the same effect on a specimen as the actual operation does on the bulk? This simple criterion becomes complicated in the case of light-fastness testing, because the exposure conditions in normal use vary so widely. For example, an evening gown during its life will invariably be exposed to much less light than a curtain fabric. It therefore follows that a light-fastness test is rarely sufficient, in itself, to satisfy all interests in the textile trade: it must inevitably be accompanied by a knowledge of what light fastness will be necessary for each class of fabric.

The establishment of such levels has always been regarded as a commercial matter and outside the scope of the Society's fastness committees, but it has been mentioned here because many cases of apparent unreliability of the test have, in fact, been due to the use of fabrics whose light fastness is too low for the purpose intended.

Unreliability of the test method can be recognised by a simple criterion. Thus a fabric whose light fastness is, say, 5 under normal test conditions may fade badly during use: if the blue wool light-fastness Standard 5 would have faded to the same extent under the same conditions of use, the test is reliable but the level is too low: if Standard 5 would have withstood the use conditions without fading badly, however, then the test is unreliable.

As the only variable factor likely to cause anomalies in testing is the effective humidity during exposure, this factor is similarly the only one which might make for unreliability of the test method.

The conditions of exposure in testing (facing south at 45°) were naturally chosen to ensure that the intensity of light falling on the pattern is the greatest which is consistent with practicability. This results in the effective humidity being the lowest which is possible during normal continuous exposure. Therefore, if the effective humidity during use is significantly higher, the test may be unreliable.

The effective humidity in use will obviously be greatest when the material is frequently exposed after having been wetted with water in the liquid state, such conditions occurring, for example, when curtains come into contact with condensation on window panes, raincoat fabrics dry out in showery weather, and dress goods are hung out to dry after washing. However, these conditions must be regarded as being outside the scope of a normal light-fastness test, as they almost come within the province of weathering. This is by no means intended to minimise the importance of testing under such conditions where appropriate, and the development of such a test is being considered by the Light Fastness Subcommittee.

When actual wetting is excluded, the highest effective humidities during exposure would be expected to occur with curtains, particularly in unheated rooms: this is because the region near a window is often the coolest part of any room and therefore the relative humidity will be greatest. Moreover, the surface heating during use is also much less than during exposure under test conditions, because the material is usually vertical and not at 45°; the amount of ventilation is usually greater, too. The highest value of all would be expected to occur in north-facing windows, as

here very little sunshine falls on the pattern, and therefore surface heating is negligible; in such cases, the effective humidity will be equal to the relative humidity of the air mass surrounding the pattern and may often exceed 80% for long periods.

It was mentioned earlier that the criterion of reliability of the test is that the pattern should fade at the same rate as the appropriate standard under use conditions as well as under test conditions. The reliability can therefore be investigated by exposing the humidity-sensitive pattern together with the standards under the critical conditions of use, covering in the normal manner so that the light fastness can be ascertained. A portion of the azoic red pattern and the standards were therefore exposed in a vertical position about I in. from the window-panes in two unheated bedrooms of the author's house in Cheshire: one room faced due south and the other due north. The results, together with those obtained from an exposure made under the test conditions in the same area at about the same time, are given in Table V.

	TA		
Time of Year	B.S. 1006	Light Fastness Domestic South	Domestic North
1954 March-May	4-5	4	3
July-Sept.	4-5	4	-
1955			
April-July	5	4-5	3-4
June Sept.	5	4-5	3-4
1955-1956			
OctApril	4-5	4-5	3-4
NovMay	4-5	4-5	3
1956			
May-June	5	4-5	4

These results show that, as far as domestic south exposures are concerned, the test conditions are reliable, because even with a pattern having very high sensitivity to humidity the maximum difference is only ½ grade.

They do suggest unreliability in the case of north window exposures, however, as here the variation is 1½-2 grades. It will be noted that on two occasions the light fastness in the north window exposure was 3, the same as that which was found when the pattern was exposed facing south in an atmosphere of 100% R.H. (Fig. 1).

Further consideration, however, shows that the significantly lower assessments obtained from north window exposure are not necessarily an indication of unreliability, for the following reason: the ultimate purpose of routine light-fastness testing is to obtain an idea of the time required for serious fading to occur in use, and therefore, providing that no pattern fades more rapidly in a north window than when exposed simultaneously facing south in the same place, the test will still be reliable.

The azoic red pattern was therefore exposed on several occasions facing south and facing north throughout the year. Sometimes the amount of fading was slightly greater facing north, but never to an extent which would be noticed by a noncolourist, and if there had not been a sharp boundary between unexposed and exposed areas it is doubtful whether a colourist could perceive the increase with certainty. It should be noted that both north and south windows were not obstructed by neighbouring trees or buildings, as the presence of these would clearly invalidate the results.

It must be pointed out that the rate of north window fading approaches that of south window fading only in cloudy countries, because the intensity of radiation from blue sky falling on a pattern exposed under glass is very low; four months' exposure to north light in Bombay during the sunny period produced only a just perceptible fade on Standard 3; four months (May-August) in the North of England produced a marked fade on Standard 6.

Consideration of these observations shows that, whenever the exposure conditions in normal use are such that the effective humidity increases, thus resulting, in the case of a sensitive pattern, in an increase in the fading rate per unit of light intensity, the intensity itself is inevitably reduced, so that the fading rate per unit of time is not significantly greater. A light fastness of 3 in a north window and 4–5 in a south window means that the fading rate of the pattern per unit of light intensity is three times as great in the north window, but the intensity of light must be less, and it would appear to be about one-third of that in a south window.

#### Conclusions

It is probable that the reproducibility of the I.S.O.-recommended Daylight Test is generally very good, no matter where or when the exposure is made, except for materials which are particularly sensitive to variations in moisture content during exposure: such patterns may be assessed as much as 2 grades higher in sunny countries than in predominantly cloudy countries such as the United Kingdom.

It is, however, neither practicable nor desirable to modify the method so that identical results will be obtained all over the world with such materials, for to do so would be to achieve reproducibility only at the expense of reliability.

A continuous daylight test provides a reliable guide to the expected resistance to fading which will occur during use even of moisture-sensitive fabrics, providing that the material is not actually wetted and provided that exposure is made in the country in which the material will be used or in a place where the climatic conditions, particularly the amount of sunshine, are similar. Exposures made in sunny countries will give optimistic assessments of such fabrics if they are to be used in cloudy countries; whilst, conversely, exposures made in cloudy countries will underestimate the actual resistance to fading shown in use.

The author realises, however, that it is not always practicable to make exposures in the country in which the material will be used or even in one of similar climatic conditions; but, fortunately, there is a possible solution to this problem. In the past, advocates of carbon-arc fading lamps have claimed that one of their

virtues was that they provided ambient conditions which were constant, unlike those occurring in daylight testing; European authorities, however, consider that the excessive ultraviolet emission of such lamps outweighs all possible virtues, and hence daylight is preferred. Now the xenon arc has an emission spectrum identical with sunlight for all practical purposes, and therefore a xenon fading lamp providing ambient conditions which could be varied to simulate those of daylight tests made in various parts of the world would solve the problem.

. . Finally, the author wishes to thank the many individuals who made exposures of sensitive materials in various places throughout the world.

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### COMMUNICATIONS

### The Light Fastness of Dyed Fibres—A Statistical Study

C. H. GILES

(with an Appendix by John C. Eaton)

About 16,000 published commercial light-fastness assessments have been examined statistically, and from the results the following general rules are formulated, which apply to most dyes and fibre-

(a) On different fibres the fastness of a dye is higher the higher the regain of the fibre, provided that this is greater than about 4% (i.e. the value for nylon). Thus dyes are usually faster to light on viscose rayon than on cotton, on cotton than on nylon, on wool than on silk, and on secondary cellulose acetate than on cellulose triacetate or nylon.

(b) The above rule is reversed with fibres having regains below about 4%, i.e. with the newer synthetic fibres: with these the fastness is usually higher on the fibre of lower regain.

(c) Fastness is often reduced by (i) solid foreign materials in the fibre, e.g. dulling agents or the resin used in crease-resist finishes; or (ii) either cationic surface active agents or powerfully hydrogen-bonding compounds, e.g. phenols or urea, used as dyeing assistants or aftertreating agents.

(d) The increase in light fastness with increase in depth of any dye is usually greater the higher the regain of the fibre: it tends to be higher with dyes which are insolubilised in the fibre than with water-soluble ones. The increase in average fastness for a whole class of dyes for a tenfold increase in depth is usually about 11 units for water-soluble, disperse, and vat dyes, and about 3 units for azoic and sulphur

The effects summarised by these general principles are considered to be the result mainly of differences in the physical form of the dyes in the fibres, chemical action being important only in a few special cases, e.g. the effect of titanium dioxide delustrant.

There is, however, a more general chemical influence which affects comparisons between certain fibres. This is the difference between the chemical nature of fading on protein and that on non-protein fibresreduction and oxidation respectively— which has some effect on the comparative fastness of a given dye on fibres of the two classes, mainly at the upper and lower ends of the scale. Thus, dyes of low fastness on a non-protein, e.g. cotton, viscose rayon, cellulose acetate, or nylon, are likely to have a fastness about one

non-protein, e.g. cotton, viscose rayon, cellulose acetate, or nylon, are likely to have a fastness about one or two grades higher on a protein, e.g. wool or silk (and perhaps Ardil fibre); and those of high fastness on a non-protein are likely to be graded one or two units lower on a protein.

Some suggestions are made of means by which light fastness might be improved; e.g. better fastness is more likely to be obtained by eliminating all small dye particles in the fibre than by encouraging larger ones to grow if small ones are also present. This is because the smaller particles tend to fade more rapidly than the larger, and therefore their fading rate determines the fastness grade of the dyed material, which is intended in the control of fading stone two soft day redeated, likely to exclude the fading rate determines the fastness grade of the dyed material, which is judged in the earlier stages of fading. Some types of dye molecule likely to exhibit high fastness are suggested.

The reliability of the fastness data is discussed. Fastness assessments given by different manufacturers for the same dye appear to agree for wool dyes, but Continental assessments for dyes on cotton are usually about one unit higher than those given in this country. This is believed to be due to longer periods of sunshine on the Continent.

#### I-Introduction

#### I. PURPOSE AND SCOPE OF PRESENT WORK

Recent investigations in this laboratory 7, 15, 20, 31, have established certain general principles explaining the changes in light fastness which a dye may exhibit if applied under various

conditions or to various fibres. The present paper describes a study of the light-fastness grading of commercial dyes, made to learn whether the principles discovered in the laboratory actually apply to all dyes on all fibres. It was hoped that a general interpretation of the mechanism of fading

might emerge, which would co-ordinate the results of earlier work in this and other laboratories, explain the origin of the many apparent anomalies in light fastness so often met in practice, and perhaps enable suggestions to be made for obtaining coloured textiles and other materials of better resistance to light. The study has included the scrutiny and the examination of the reliability of about 24,000 individual assessments of fastness published by the manufacturers of dyes, and from these about 16,000 have been used in a variety of tests and comparisons, which have been statistically checked. The conclusion reached is that the broad principles established earlier do actually apply to all textile dyeings, but other factors affecting fastness are also revealed. Certain general suggestions for improving light fastness are made.

In the previous experimental work it was shown that fading on protein fibres is of a different chemical nature from fading on all other fibres <sup>15, 29</sup>. It was also shown that not only the chemical nature but also the physical form of the dye and that of the fibres are of importance in their effects on fading rate. The method of application of the dye and the porosity of the fibre can affect the light fastness of a dye at any concentration, and also the rate at which the fastness improves with increase in depth of dyeing <sup>7</sup>.

The present study covers the influence of the chemical and the physical factors upon fading.

### 2. RELIABILITY OF THE DATA

It is of course essential first to assess the reliability of the light-fastness data which it is proposed to study. This is discussed in detail later (pp. 152 and 154–155), but the conclusions reached may be

referred to briefly here.

Much of this investigation is based upon the use of averages of large numbers of results, and the averaging process does of course "iron out" many irregularities arising during testing, but not all. It will be shown that fastness gradings for dyes on fibres other than wool are liable to differ between manufacturers on account of variations in atmospheric conditions at the time of testing. It is considered that comparisons of fastness are most reliable when they are made between- (a) dyes on wool; (b) classes consisting of large numbers of dyes which have probably been tested over a period of years; (c) figures for the same dye on two fibres quoted in the same text, as these have probably been obtained by simultaneous exposures. In all other cases, the best test of the reliability of comparisons is their consistency amongst themselves. It will in fact be shown that on this basis the comparisons are valid.

#### 3. THE STATE OF DYES IN THE FIBRE

It is proposed to show that very many of the phenomena of light fastness are best interpreted on the assumption that dyes, whether water-soluble or not, are usually present in fibres as heterogeneous collections of particles ranging from molecularly dispersed material to aggregates or microcrystals of various sizes. The bearing of this hypothesis upon light fastness has already been discussed? The more the dye is aggregated, the

higher the light fastness becomes, because the surface area of a given amount of dye becomes less as the particle size increases, and fading is believed to take place mainly at the surface. Aggregates can develop more readily in fibres of low than in those of high crystallinity, so that there should be a general tendency for light fastness to be better on the more porous fibres.

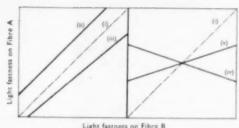
Singularly little direct evidence is available upon the state of water-soluble dyes in normally dyed fibres. Astbury and Dawson 4 detected submicroscopic crystals of some anthraquinone dyes in wool, by X-ray diffraction techniques. Morton 44 deduced, by an interesting series of tests with polarised light, that certain direct dyes in a highly crystalline cellulosic fibre (ramie) are oriented as single molecules attached to the cellulose chains. The results do not, however, rule out the possibility that some aggregated dye also may be present. Astbury and Dawson used dry wool in their tests, so that it is not clear whether the dye crystals in the fibre were formed during the dyeing process or afterwards during drying. It has been suggested previously 7 that the origin of such aggregates may lie in the disturbance of the complex fibre-dye-water equilibria on drying the fibre. Recent spectrophotometric tests in this laboratory, however, have shown that at least in some cases (basic dyes in gelatin) aggregates are formed during dyeing, and tend. rather to break down on subsequent drying if heat is used (Campbell, private communication).

Nothing appears to be known of the change in the state of the molecular chains of fibres when they are dyed. Even the smallest dye molecule is apparently much larger than the largest pores in dry fibres, so that the molecular chains in dry fibres must become distorted in presence of dye molecules, whether these are arranged singly or as aggregates. The distortion might lead to an increase in compactness of fibre structure near the dye, and thus perhaps to a decrease in accessibility to air and moisture (cf. Baxter et al.7, System C, Fig. 1). It might be thought that this could produce effects similar to those expected from aggregation, even if the dye is entirely molecularly dispersed, but most of the evidence of experimental work on films and fibres dyed with a variety of types of dye 7, 31, 32 (also Baxter, private communication) suggests that dyes applied normally are more accessible to these agencies when molecularly dispersed than when aggregated. It appears that oxygen and water vapour can usually penetrate to the sites of dye molecules or crystals held between the molecular chains of a fibre substrate, though in the more highly crystalline synthetic fibres, e.g. Terylene and Orlon, penetration is much more difficult than it is in less organised structures.

### II— Chemical Influences affecting Light Fastness

I. GENERAL DIFFERENCE BETWEEN PROTEINS AND NON-PROTEINS

It has been shown in a previous paper <sup>20</sup> that fading on proteins is probably a process of reduction



(a)—Both fibres protein or both (b)—One protein and on non-protein fibre

(i) No difference between Fibres A and B in effect on fastness
(ii) Dyes on Fibre A tend to have higher fastness than on Fibre B
(iii) Dyes on Fibre A tend to have lower fastness than on Fibre B
(iv) and (v) Curves have negative or much less positive slope on account of difference in chemical nature of fading on Fibres A and B

Fig. 1 — Theoretical Comparisons of Light Fastness of Dyes on Pairs of Fibres

of the dye, whereas on all other fibres, including e.g. all cellulosic fibres and nylon, it is an oxidation. Because reduction and oxidation are chemically opposite in nature, the sequence in a series of dyes arranged in order of increasing fastness on a protein fibre should be opposite to that on a non-protein. In other words, if the dyes A. B. C. D, E, and F increase in fastness in that order on a protein fibre, say wool, the order on a nonprotein, say nylon, should be F, E, D, C, B, A. A few selected series of dyes tested in the laboratory did in fact show this reversal 20. The present test was made to determine whether the same difference in behaviour applies to all dyes and fibres. For convenience, the difference between protein and non-protein fibres is referred to below as being a difference of "photochemical class". The test is made as follows

The fastness on one fibre of each individual dye of a series, all of the same dyeing class, is plotted against its fastness on another fibre. This is done for pairs of fibres which are both non-proteins or both proteins, and also for other pairs which consist of one of each class. Fig. 1 shows hypothetical curves for typical conditions which might be encountered in this type of comparison, thus—

### (a) Both Fibres Non-protein or Both Protein

If each dye has exactly the same fastness on the two fibres, then, of course, line (i) will be obtained, which is for convenience referred to below as the "equivalence line". This condition is unlikely to occur, and it is more to be expected that one fibre will tend to alter the physical conditions in such a manner as to give consistently higher or lower fastness than the other. This effect would produce either line (ii) or (iii). In one case it is supposed that the difference is constant over the whole fastness range, in the other that it changes slightly from one end of the range to the other. The causes of these differences are discussed on pp. 137-138 and 144-146. An important point to note at this stage, however, is that, unless the nature of the physical influence responsible for the differences between the fibres is different at the two ends of the fastness scale, the whole curve should lie either below or above the equivalence line and should not intersect it.

### (b) One Fibre Protein and One Non-protein

If all the dyes tested are of the same chemical class in most cases there should be a reversal of slope of the curve, for the reasons already given. It is unlikely that the difference between the dyes of lowest and highest fastness will still remain the same on both fibres, so that a curve (iv) might result which is flatter, but has a negative slope. The dyes examined in the earlier work <sup>20</sup> mostly behave in this manner when examined series by series, with the exception of two pairs, which did not reverse their sequence; the reasons for this were discussed.

Dyes comprising a whole dyeing class are unlikely to form a single chemical series except in special cases, e.g. certain metal-complex dyes. Each dyeing class may contain members of a variety of chemical series, most of which, if they could be identified, might be found to reverse in sequence amongst themselves, though some might not. The whole class, however, would not give a line of negative slope, and a curve of much less positive slope (v), with considerable scatter of points about it, seems likely to be encountered. In both cases, however, viz. whether a single series or a collection of series is under test, the line should still intersect the equivalence line (i).

The effects of aggregation also could prevent a negative slope from occurring, in the following way. Let it be assumed that the higher is the fastness of a dye in a particular series, the greater is its degree of aggregation. Take, then, a hypothetical case in which two dyes, A and B, would have fastness of 1 and 4 respectively on a fibre X, if they were both completely molecularly dispersed. Their fastness order will reverse when they are applied to a fibre Y of the opposite photochemical class, though the numerical difference between their gradings would not necessarily be the sameit might be either greater or less. Suppose it is less, and the dyes are rated at 3 and 2, so that the plot for these dyes has a negative slope. But now suppose that B is actually considerably aggregated on both fibres, so much so that its fastness is

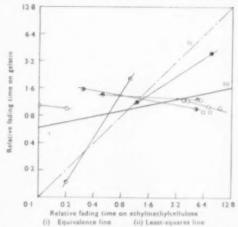


Fig. 2 — Comparison of Light Fastness of Six Different Series of Dyes by Spectrophotometric Measurement on Protein and Non-protein Substrates<sup>20</sup>

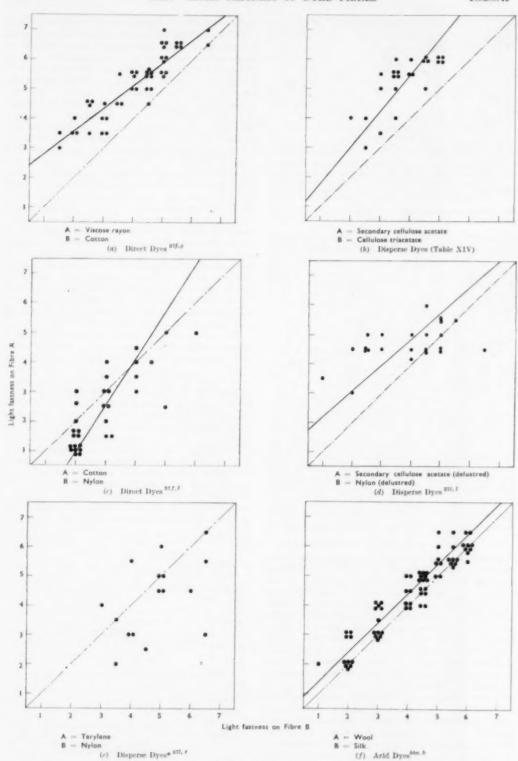


Fig. 3. (a-f)—Comparison of Light Fastness of Individual Dyes on Pairs of Fibres. • In (e) and (l) the least-squares lines have low significance and are not shown

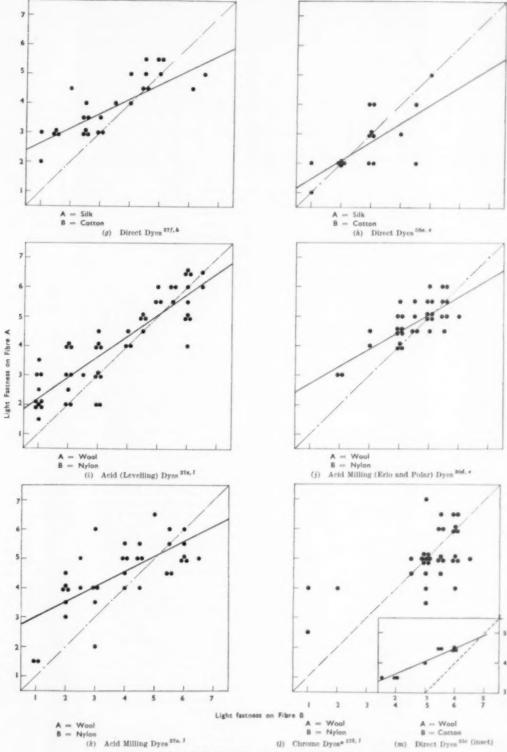


Fig. 3 (g-m)— Comparison of Light Fastness of Individual Dyes on Pairs of Fibres • In (e) and (l) the least-squares lines have low significance and are not shown

thereby increased by 3 units, whereas A remains unaggregated. The position then is that on transfer from fibre X to Y dye A increases in fastness from 1 to 3 and dye B decreases from 7 to 5. The slope of the plot is then still positive, but it cuts the equivalence line (cf. v, Fig. 1).

The hypothesis was first tested by plotting experimental data obtained in this laboratory cf. 20; the logarithms of relative fading times (shown below to be equivalent to light-fastness gradings) of some dye series on a protein (gelatin) and on a non-protein (ethylmethylcellulose) respectively were plotted against relative concentration. The best straight line (by the method of least squares) through all the points has the anticipated reduced slope (Fig. 2).

The test was next extended to classes of commercial dyes, fastness grades on two fibres being plotted against each other.

Typical curves showing such comparisons made by the use of fastness grades for several classes of dyes on various pairs of fibres are shown in Fig. 3 and 4. Fig. 3(a-f) are for pairs which are both of the same photochemical class, either non-protein (Fig. 3a-e) or protein (Fig. 3f). The calculated least-squares lines are shown, and the equivalence line for comparison.

In all except one of the comparisons made between two fibres of the same class there is a fairly constant difference in fastness over the whole range, and none of the curves crosses the equivalence line. The exception is the plot for direct dyes on nylon and cotton (Fig. 3c), which is anomalous probably because nylon affects fastness by different mechanisms at the two ends of the scale; this is discussed below.

With two possible exceptions the plots for comparisons made between two fibres of different

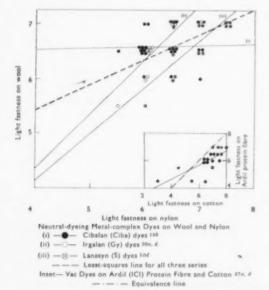


Fig. 4— Comparison of Light-fastness Assessments of Dyes on Protein and Non-protein Fibres

classes (Fig. 3g-l and 4) show the predicted change of slope, and cross the equivalence line. The exceptions are the Irgalan and Lanasyn classes of neutral-dyeing metal-complex dyes on wool and nylon (Fig. 4). The Irgalan dyes give a line parallel to the equivalence line, and the Lanasyn dyes one which crosses it at a very small angle, though in the predicted direction. The Cibalan dyes show a more marked slope change than any other class examined. All these dyes are unsulphonated 2:1 dye-metal complexes. interpretation of the differences between them must be that the nature of the solubilising groups is different in the three classes. The Irgalan dyes (which contain substituted sulphonyl groups as solubilising radicals 51), and perhaps the Lanasyn dyes also, are presumably reduced and oxidised at different centres in the molecule (cf. the Rhodamines 20). This seems a more likely explanation of the curves they give than to suppose that the reduction mechanism on proteins ceases to operate for these classes.

It is interesting that the data for vat dyes on cellulose fibres and Ardil protein fibre (Fig. 4) show change of slope, though the figures do not cover enough of the range to have much significance. This is the only evidence that Ardil behaves as a normal protein fibre in fading.

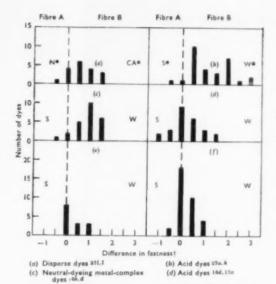
The general conclusion drawn from all these comparisons is that the difference in chemical nature of fading on proteins and non-proteins probably persists throughout all classes of dyes and fibres.

#### 2. SPECIFIC DIFFERENCES BETWEEN SUBSTRATES OF THE SAME PHOTOCHEMICAL CLASS

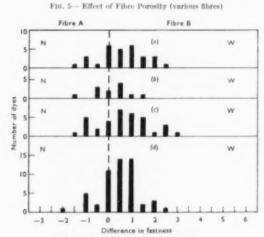
### (a) Dyes promoting Photochemical Degradation of Fibres

The effects discussed in § II.1 above apply apparently to all dyes. The difference between the action of proteins and of non-proteins in fading is thus a quite general one. There are, however, some chemical effects which are more specific and are observed only in the fading of particular dyes on certain fibres. These include the well known accelerated tendering of fibres by light in the presence of so called "active" dyes, at one time believed to be confined to vat-dyed cellulosic fibres, but now known to occur with several other classes of dyes, e.g. basic, azoic, disperse, and sulphur dyes, and with other fibres, e.g. nylon and silk. For a summary of investigations upon this subject Egerton's survey 27 should be consulted. The cause is believed to be the degradative effect of hydrogen peroxide released by the action of light on the active dye 26, and the better known forms in which this is manifested are either (i) tendering of the fibre without any accelerated fading of the dye itself, or (ii) accelerated fading of a second inactive dye present in admixture with the active one.

Neither of these changes would affect the data collected here. Egerton <sup>25</sup>, however, has shown that this type of photochemical reaction can in some cases promote the fading of dyes on nylon, and the



(e) Acid (levelling) dyes 600,6 (f) Acid (milling) dyes 50a, 5



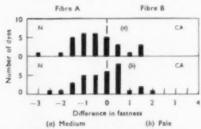
(a) Mordant dyes (Eriochrome) 395. 204

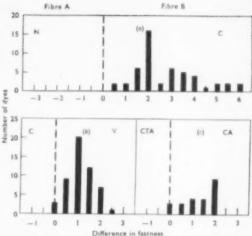
(b) Acid-dyeing metal-complex dyes 37c. I

(c) Acid milling dyes 37s. I

(d) Acid levelling dyes 87a, I

Fig. 7— Effect of Fibre Porosity (Nylon and Wool)

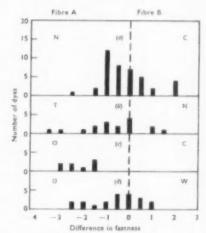




(a) Vat dyes 37d, I

(b) Direct dyes 87% ø

(c) Disperse dyes (Table XIV) Fig. 6— Effect of Fibre Porosity (various fibres)



(a) Ordinary direct dyes 27f. (

(b) Disperse dyes (heavy dyeings) 271. s

(c) Basic dyes 570, 0

(d) Acid dyes 27a. a

Fig. 8— Effect of Low Regain in Various Fibres

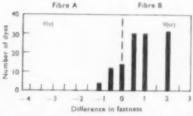


Fig. 9-- Effect of Depth of Disperse Dyes on Fibres of Low Regain 166 Fig. 10-- Effect of Crease-resist Treatment on Ordinary Direct Dyes 166

Fig. 5-10-- Frequency polygons showing the Influence of Regain, etc. on Light Fastness on a Variety of Fibres

· Abbreviations for fibres-

(lations for units)

C = Cotton

CA = Cellulose acetate (secondary)

CTA = Cellulose triacetate

N = Nylon

O = Otlon

S = Silk

T = Terylene

V = Viscose rayon
W = Wool
u = untreated
cr = crease-resist treated.
The horizontal axis shows the difference in fastness; a positive value means that the fastness is higher by the given amount on the fibre (B) named on the right than it is on the fibre (A) named on the left; a negative value means that the fastness is higher on A than on B.

Table I

Distribution of Light-fastness Differences in Identical Dye Series on Pairs of Fibres, etc.

(No. of dyes having the specified difference in fastness grading on Fibre B compared with Fibre A)

Ref.   States Chemical   Percent   States   St	Difference in factors grading   Class   Clas
Figures   Parish	Figures grading   Class   CHEMICALEX LORNITION   CHEMICALEX LORNITIO
Figures Chemically Decrease   Figures   Figures Chemically Decrease   Figures   Figu	Ref.
Ref.	Fibres   Strate   Fibres   Strate   Fibres   F
SCHEMICALLY IDENTICAL OR SIMILAR  CHEMICALLY IDENTICAL OR SIMILAR  CHEMICAL ONE PROTEIN, ONE NON-PROTEIN  CHEMICAL ONE PROTEIN  CHEMIC	S 2-3 2 1-2 1 0-1 Nil 0-1 1 1-2 2 2-3 3  CHEMICALLY IDENTICAL OR SIMILAR  OF THE PROPERTY OF T
2-3 2 1-2 1 0-1 Nil 0-1 1 1-2 2 2 2-3	2-3 2 1-2 1 0-1 Nil 0-1 1 1-2 2 2-3 3
L-Both Non-Protein  1	Leented OR Similar  Los 1-2 1 0-1 Nil 0-1 1 1-2 2-3 3 3 4 4 4 1 1 1 1 1 1 1 1 1 1 1 1 1 1
OR SIMILAR  4 0 0 1 1 1-2 2 2-3  1 0-1 Nil 0-1 1 1-2 2 2-3  1 3 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5	OR SIMILAR  1 0-1 Nil 0-1 1-2 2 2-3 3 3 4 4 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1
OR SIMILAR  1 0-1 Nil 0-1 1 1-2 2 2-3  4 0 0 2 4 11 11 7-1 Increase  1 0 1 0 2 0 1 1 1 1 2 2 2 3  1 0 0 0 0 0 0 0 1 1 1 1 1 1 1 1 1 1 1	OR SIMILAR  1 0-1 Nil  4 0 0 4 0 0 1 1-2 2 2-3 3 3 4 4 5 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1
Nil 12   22   23   24   10   11   11   12   25   25   25   25   25	Nii 1-2   2   2   2   2   2   2   2   2   2
11	11 11 11 11 11 11 11 11 11 11 11 11 11
1	1
1.   1.   2. 4.   2. 0. 0. 0. 0.   0.   0.   0.   0.	1-   2 2 4-1   20 10 20 10 10 10 4 10 0 4 10 10 10 10 10 10 10 10 10 10 10 10 10
	10 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1
	**    -
00    -	
	1 10 11111 1 1 111111111111111111111111

Nylon					Secondary c (pigment- delustred)	Viscose rayon (crease- (un	resisted)										(Levogen WW)	(Fixanol	PN §)	Visor	WW)	Fixanol PN				Viscose rayon	nrea process)
Wool					Secondary cellulose acetate pigment. (bright) elustred)	ayon (untreated)										Cotton	(untreated)	(untreated)		Viscose rayon	( marrie Constant)	(Fixanol PN§) (untreated)				Wool (Amed)	
Neutral-dyeing metal-complex		Chrome			Disperse	Direct (ordinary)	(Diphenyl)	(Dipnenyl rast)	16	(light-fast,	(light-fast)		Vat	(anthraquinone) (indigoid)			Direct	(ordinary)	(light-fast)	( condition in the control	(Creation 2)	(ordinary)	(hght-fast)	(Teyl)		Acid milling	Direct (light-fast)
1	1	1	1		1	1		11	1	1	1	1	1	1	D		1	1	!			1	1	I		1	1
(100)	(CDC1)	(874)	1		(321)	(25e)	1991	(376)	(906)	(290)	(378)	(304)	(37e)	(324)	DYEINGS UNI		î.	(37.67)	(55.50)	/25a/	(38)	(27.2)	(327)	(37.57)	DYES	(970)	(418)
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1 .	-	10	Ç1		ADDONA 	01	•	* 0	30	1	0	10	1	1	WITH (		23	1	1-	00	200	19	10	<b>6</b> 1	WITH (	01	0
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wy I	-	11	1-		60	00		200	14	17	13	×	99	1	AGENTS		101	63	50			į E-	k K	1		Q1	10
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1	1	63	1		1	1		*		4	01	4	-	1			1	1	1					1		10	1
1	1	0	1		1	1		1		0	-	1	1	1			1	1	1					1		65	1
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1	1	1	-		1	1		1		1	1	1	I	1			1	J	1					1		523	E
			7						1	- 1		. 1	1				1	1	1		1			i		-	1

\* H = Heavy; M = Medium; P = Pale.

† Private communication.

‡ Decrease > 3 grades.

‡ Private (CDC) (estytypridinium bromide) also reduces light fastness considerably. Out of 130 direct dyes, 105 are reduced in fastness in various degrees, 22 are unaffected, and 3 are improved (ICD).

few disperse dyes which are most markedly less fast on this fibre than on cellulose acetate, e.g. Duranol Brilliant Yellow 6G (ICI), are in fact very effective in promoting the tendering of nylon by light. This can explain a few of the lower figures for light fastness of disperse dyes on nylon compared with cellulose acetate, but by no means all, because the difference in fastness persists in many dyes which do not promote the tendering of nylon, and in some which actually behave as protective agents <sup>25</sup>.

All the vat dyes which accelerate the tendering of cellulosic fibres by light are said to have very low light fastness on nylon<sup>23</sup>. It appears, therefore, that accelerated tendering of this fibre is generally accompanied by abnormal photochemical destruction of the dye.

### (b) Chemical Effect arising in the Crease-resist Process

Fabrics treated by the urea-formaldehyde crease-resist process always retain a small amount of free formaldehyde, which, being a reducing agent, may tend to retard the oxidative fading of the dye. In this case, however, another independent factor must be considered, physical in origin and acting to accelerate fading, namely the decrease in the pore-size of the fibre. The results which follow the concurrent action of these two factors will be discussed in § III.4 below.

### (c) Fastness of Dyes on Anodised Aluminium

Dyes have higher fastness on films anodised in sulphuric acid than on the less porous films anodised in chromic acid 7. 37dd (Table I). It was previously considered that the difference is due to the dyes being able to form larger aggregates in the sulphuric-acid film 7, but following later investigations of the mechanism of the dyeing of anodised aluminium\*, it is thought that the state of aggregation may be very low in both films, and that the fastness difference may be due partly to a chemical effect. The chromic-acid film is known to contain some chromium from the electrolyte, which, though small in relation to the total weight of film (ca. 0.1%) 33, is of the same order as the weight of dye faded in testing for fastness. Fading on aluminium is likely to be oxidative 15, and therefore an oxidising agent should act as an accelerator, so that the trace of chromium could reduce the light fastness of the dyed film (cf. the opposite effect of formaldehyde in cotton, p. 138). Even on this film, however, and still more so on the sulphuric-acid film, many dyes have high light fastness, which may be attributed to blockage of the pores to vapour diffusion by the growth of metal hydroxide which follows the "sealing" with steam or boiling water normally given after dyeingt; some sealing may even occur by the action of hot water during the actual dyeing operation. Improved light fastness with increase in dye concentration in this system would then be due to more effective sealing.

### III— Physical Influences affecting Light Fastness

#### 1. GENERAL CONSIDERATIONS

It is seen from the discussion above that light fastness is influenced by the chemical reactivity of the dye, but not to a great extent. Baxter et al. <sup>7</sup> found, however, that the physical state of a dye can have a considerable effect on fastness, and in the present section the conclusions of their work will be tested against as many as possible of the published fastness assessments on fibres. Some additional hypotheses will also be examined.

One of the most satisfactory ways of making the required tests is to compare the distribution of fastness differences for a whole class of dyes on a pair of fibres. The results can conveniently be expressed tabularly or diagrammatically, by frequency polygons. Both methods are adopted here. Some results are shown diagrammatically in Fig. 5-10 and others are tabulated (Table I). These comparisons are made in each individual case between the same apparent depth of colour on the two fibres. (The depth may appear the same to the eye, but the actual amount of dye present may, of course, be slightly different on the two fibres. It seems extremely unlikely, however, that any error introduced by this factor can affect the results significantly. Even a difference as great as 100% between the amounts of dye on the two fibres, which seems considerably more than will occur in practice, would produce a difference in fastness of only 0.3-0.4 grade (cf. § IV and Table VI below), and this is much less than most of the differences observed.) A detailed discussion follows.

#### 2. COMPARISONS BETWEEN UNTREATED FIBRES OF DIFFERENT POROSITY

As a first approximation it may be assumed that the water-absorption figures lie in the same order as the porosity or amorphous: crystalline ratio of the fibre (Table II). It will then be seen from the

TABLE II
Water-absorption Data (%) for Fibres

Fibre			Vapour (regain)	Liquid (imbibition)
Terylene			0.4	5
Orion (type 42, stap	ple fib	re)	1.5	_
6,6-Nylon (drawn)	***		4	17
Cellulose triacetate		117	4.5	16-5
Cellulose acetate (se	conda	ry)	6	30
Cotton*	***	***	8-5	48
Silk			11	48
Viscose rayon*		2 0 2	11	100
Wool			16	50

Some rate experiments <sup>48</sup> appear to indicate a smaller pore size for viscose rayon than for cotton. This must be the result of viscose rayon having a "skin" of more highly oriented cellulose <sup>68</sup>, <sup>57</sup>.

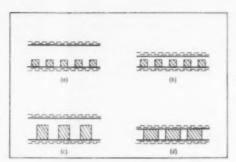
data that the systems can be classified into two groups—

### (a) All Dyes having the Same or Higher Fastness on the More Porous Fibre

These systems (Table IIIa) conform with those studied earlier?. The fastness improvement may be said to occur through "promotion of aggregation".

<sup>·</sup> Stewart, private communication,

t Tests in this laboratory (Catchpole, private communication) have shown that scaling does improve light fastness,



o, b Small aggregates unaffected c, d Large aggregates restricted in growth

Fig. 11— Illustration of Effect of Decreased Fibre Porosity on Dyes with Small and Large Aggregates

The fact that some dyes are not improved, but retain the same fastness when applied to a more porous fibre, is not necessarily contrary to the hypothesis already outlined (p. 128) for the following reason. If the particles in which the dye

### TABLE III

#### Summary of Differences in Fastness of Dyes on Two Substrates

Dye Class	Subs	trates
	A	В
(ai) All Dyes	OF HIGHER POROSE	NESS ON SUBSTRATE
Azoic	Cellulose acetate (secondary)	Viscose rayon
Direct (ordinary)	Cotton	Wool
Direct (Icyl)	Nylon	Cotton
Direct (light-fast)	Nylon	Cotton
Disperse	Cellulose acetate	(secondary)
	(dull)	(bright)
Disperse	Cellulose triacetate	Cellulose acetate (secondary)
Vat	Nylon	Cotton
	OF DYES HAVE HE	OSITY
Acid wool, mordant, metal-comple	Silk x	Wool
Acid wool, mordant, metal-comple	Nylon	Wool
Direct (light-fast)	Nylon	Cotton
Direct	Viscose r	ayon
(light-fast)	(crease-resisted)	(untreated)
	OF DYES HAVE LO	
Direct (ordinary)	Nylon	Cotton
Direct	Viscose r	ayon
(ordinary)	(crease-resisted)	(untreated)
Acid wool	Orlon	Wool
Disperse	Orlon	Cellulose scetate (secondary)
Disperse	Terylene	Cellulose acetate (secondary)

Terylene

Orlon

(bii) ALL DYES HAVE HIGHER FASTNESS ON FIBRE OF LOWER POROSITY

Disperse

Basic

Nylon

Cotton

normally occurs are smaller than the pores of the less porous fibre, they may have the same dimensions, and thus the same fastness, in both fibres. A simple diagram (Fig. 11) illustrates this.

### (b) Most Dyes having Lower Fastness on the More Porous Fibre

One possible explanation for this behaviour (Table III b) is that the dyes which appear to be anomalous may have been tested on the two fibres at different times, when the atmospheric conditions were not the same. If, however, there is a tendency for many dyes in a series to show this effect, and especially if it is seen in independent assessments made by different manufacturers, it is much more reasonable to seek the cause in some property of the dye-fibre system itself, which opposes the effect of increased aggregate size. This property could be either chemical or physical. The only pair of untreated fibres, however, in which the anomalies might be due to an opposing chemical effect consists of wool and silk. The protein fibres are probably the only fibres in which the fibre molecules themselves play a part in the fading reaction of the dye <sup>15, 30</sup>, so that chemical differences between silk and wool might cause some dyes to be photochemically destroyed more slowly on the former fibre even though they were present as smaller particles.

Most of the anomalies under this heading, however, appear to be due to a physical effect, very likely the retardation of diffusion of moisture towards the dye. This retardation can be attributed either to a naturally highly crystalline fibre structure (e.g. in Orlon or Terylene), or more probably to the growth of large dye aggregates, which seal off parts of the inner fibre structure\*, or to the two effects combined. This explanation appears to cover the behaviour of many of the series of dyes on cellulose triacetate, nylon, Orlon, and Terylene; and perhaps it may apply also to a few dyes on silk. This type of increase in light fastness may be referred to as a "diffusion-restriction" effect. (Not only a reduced rate of moisture diffusion into the fibre, but also a reduced rate of ingress of oxygen and of egress of decomposition products from the faded dye, may contribute to the improvement in fastness.)

Obviously, the diffusion-restriction effect should be more noticeable with dyes of low than with those of high fastness, for the former will require to be supplied with moisture more rapidly if the fading reaction is to be maintained at its normal pace. This might explain why amongst the direct dyes only those of lowest fastness on cotton have better fastness on nylon than on cotton. The normal diffusion rate in nylon is adequate to maintain fading of the more slowly decomposed dyes, so that the relative fastness of these dyes on the two

<sup>•</sup> Some preliminary measurements (Elder, private communication) do not disclose any significant difference between dyed and undyed Terylene fibres in either the rate of absorption of water vapour or the amount adsorbed at equilibrium (in wool there is usually a marked difference). If this is confirmed by more detailed work, it would suggest that at least some of the absorbed dye is located in regions of the fibre to which water does not readily penetrate. This could account for the diffusion-restriction fastness effect (cf. the Procion dyes, pp. 139–140).

fibres is then controlled only by the "promotion of aggregation" effect.

### 3. VAT DYES AND CHROME DYES ON NYLON

Vat dyes generally have a much lower fastness on nylon than on cotton, attributed to their much smaller particle size on the former fibre <sup>7, 23</sup>. These dyes, in their oxidised state, are present in the form of water-insoluble crystals and so cannot be attached to the polar groups in either fibre. These groups are therefore able freely to adsorb water and an adequate supply of moisture is available for fading in both fibres. The fading of the dyes on nylon can then proceed readily\*, and the maximum difference between fastness on that fibre and on cotton is achieved.

The other dyes used on nylon, viz. acid, direct, disperse, and metal-complex dyes, differ from the vat dyes in being attached to the nylon fibre itself, either by electrovalent linkages or by hydrogen bonds, through polar groups in the fibre. This will reduce the ability of the polar groups to maintain the rate of diffusion of moisture to the site of the fading reaction.

Chrome dyes of two brands show some tendency to be less fast on nylon than on wool (Table I), but dyes of the same class made by another manufacturer show a more noticeable tendency (Fig 7). There is a difference in the method of chroming used in these cases. The first two brands are applied by use of a reducing agent (sodium thiosulphate) in the chroming stage, which effects rapid reduction of the potassium dichromate to the mordant (chromic salt) and avoids the prolonged boiling in an acid solution otherwise necessary. The other brand is dyed by a prolonged boil in acid. Presumably in the first two cases some of the polar groups in the fibre not occupied by dye are partially occupied by the excess chromic cations and diffusion of moisture is thereby retarded, fading rates being noticeably reduced, but not quite enough to make the dyes faster than they are on wool. When the prolonged boiling treatment is used, without reducing agent, not only may extra polar centres be developed by hydrolysis of amide linkages, but those already present will be less masked by excess chromic cations. Thus the diffusion of moisture will not be retarded, and fading will proceed more readily. This explains why the lower fastness compared with wool is more marked.

#### 4. SOLID ADJUVANTS IN FIBRES

By partially filling some of the vacant space in fibres in which dye aggregates would otherwise grow, solid adjuvants should tend to cause a reduction in light fastness. A little information is available on three systems of this nature—(a) cellulose acetate with titanium dioxide delustrant, (b) crease-resisted viscose rayon†, and (c) weighted silk.

The data for these systems show that blockage of pores in a fibre or film cf. 7 with solid matter other

than dye tends to reduce the fastness to light of the dyed material. The evidence is quantitative in cases (a) and (b) (cf. Fig. 10 and Table I). In case (c) it is confined to the statement that the light fastness decreases with increase in the amount of weighting material 37j.

There is, of course, the possibility that titanium dioxide used as a delustrant may affect the rate of destruction of dye by chemical as well as physical means. Indeed, this compound is known to sensitise the accelerated tendering of cellulose by vat dyes, and its effect upon the fading of other dyes has previously been assumed to be of the same nature. Whether chemical action takes place or not, however, the physical effect must be operative.

The crease-resisted fibre system has a more complex effect. Some direct dyes have lower fastness on the treated fibre. These are mainly the dyes of high light fastness, which suggests that their good fastness results from the formation of large aggregates; these are restricted in growth by the resin in the pores of the treated fibre.

Some free formaldehyde (ca. 0·05%) is present, however, in all crease-resisted fabrics, and this, being a reducing agent, will tend to retard fading, which on cellulose is an oxidation process (cf. § II,2(b), above). It follows that dyes which tend to be molecularly dispersed rather than aggregated, e.g. normal direct dyes, which are largely oriented as a monolayer in cellulose 4, will be little affected by a decrease in pore size, and may indeed be improved in fastness by the chemical action of formaldehyde. In confirmation, it is seen (Table I) that the ordinary direct dyes, of low light fastness, do in fact have mainly improved fastness on crease-resisted fibre.

The crease-resist treatment is applied to the already dyed fabric. It is a wet process, so that water-soluble dyes will be able to change their physical form during its operation; this accounts for the postulated reduction in size of particles of the fast direct dyes. Dyes which are insolubilised after dyeing, and therefore cannot change their form so readily, are unlikely to become less fast, but might be improved (by action of the formaldehyde). In fact, the two insoluble classes for which data are available, viz. vat dyes (Table I) and Alcian dyes 37cc, are almost entirely unaffected, or are slightly improved in fastness by the treatment.

### 5. ACTION OF SURFACE-ACTIVE AGENTS

Cationic surface-active agents assist the spreading of anionic dyes in monolayers on the surface of water <sup>31</sup>. It is also well known that they often cause a reduction in light fastness when used to aftertreat dyes, e.g. direct dyes. The two effects are almost certainly connected, the reduction in light fastness being caused by the aftertreated dye in the fibre including a higher proportion of monolayer, with a resulting increased accessibility to moisture. An earlier paper reported quantitative tests showing the increased fading rate of direct dyes in presence of Fixanol C (ICI) (cetyl-pyridinium bromide) <sup>7</sup>.

Light-fastness figures for series of direct dyes aftertreated with two proprietary cationic agents

 $<sup>^{\</sup>bullet}$  Some of these dyes fade rapidly on nylon because of accelerative tendering effects  $^{18}.$ 

<sup>?</sup> Preston and Tawde <sup>47</sup> have found lower regains for crease-resisted than for untreated viscose rayon.

TABLE IV

Effect of Carriers on Daylight Fastness of Disperse Dyes on Terylene

						Suc a marriage or mark	and and an arrangement	
Carrier	***	400	***	***	471	Tumescal PH (ICI)	Tumescal OP (ICI)	Tumescal D (ICI)
							HO	
Formula	***		***	***	***	Но-( )-он	$\langle \rangle \langle \rangle$	()-()
No. of dyes with	reduced	light	fastness	417	***	14	11	0
No. unaffected	1117	***	***	***	***	6	9	20
Mean reduction is	fastner	se of a	ffected d	von n	rades	1.3	0.9	

show that these agents have a decided tendency to reduce the light fastness (Table I).

### 6. EFFECT OF HYDROGEN-BONDING AGENTS

Powerful hydrogen-bonding agents, e.g. phenol and urea, considerably reduce the aggregation of dyes in solution. They might be expected to do the same in the fibre, thus causing a reduction in light fastness. This has in fact been demonstrated in a few cases in the laboratory\*, and a study of the present light-fastness data also reveals evidence of the effect, for phenols, and sometimes urea, employed as aids in specialised forms of dyeing, appear to reduce light fastness.

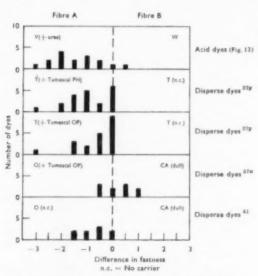


Fig. 12 -- Effect of Hydrogen-bonding Agents on Light Fastness

Both o- and p-phenylphenol are used as "carriers" to assist the dyeing of Terylene with disperse dyes. The corresponding compound without a phenolic group, viz. diphenyl, is also used. Examination of light-fastness figures (Fig. 12 and Table IV) shows that the two phenols have a marked effect in accelerating fading, but diphenyl has no effect. This is not because it is unabsorbed by the fibre, as all three compounds are in fact very substantive <sup>cf. 5p</sup>. Moreover, the p-hydroxy compound has a more pronounced action than the ortho compound. This would be expected if the cause is hydrogen bonding between the phenolic group and the dye molecule, because the ortho

group is less favourably placed to take part in such bonding.

Certain acid wool dyes can be applied to viscose rayon to give wash-fast dyeings, by a printing process employing a printing paste containing urea. Suitable dyes are all colloidal in nature, and Meitner 43 has proved that the urea breaks down their aggregates and allows them to diffuse more readily into the fibre under the steaming conditions employed. A subsequent washing treatment removes excess urea. This, and the tendency to re-aggregate when the temperature returns to normal, cause the dye to be "trapped" in the fibre, so that it cannot be readily washed out. Some urea must resist the rinsing process, however, and remain in the form of a hydrogen-bond complex with the dye, resulting in a permanent reduction of aggregation, which should be evident in lower light fastness.

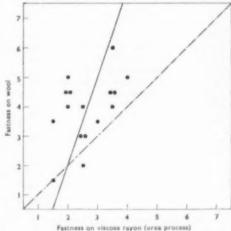


Fig. 13—Comparison of Light-fastness Assessment of Acid Dyes on Wool and on Viscose Rayon (Urea Process) <sup>279</sup>

Fig. 13 and Table I illustrate the change of fastness of some acid wool dyes applied by this method in comparison with their normal fastness on wool. It is obvious that urea does markedly reduce the fastness of acid dyes. It has a similar, but less noticeable, effect on direct dyes <sup>37</sup>; the reduction in its effect may be due to competition by the fibre, which will be greater with a substantive dye, or to more powerful intermolecular aggregating forces.

### 7. DYES WHICH REACT WITH THE FIBRE

The recent introduction of the Procion dyes 37ce, which are said to combine chemically with the

Phenol reduces aggregation and fastness; urea sometimes does this, and sometimes has the reverse effect (Campbell, personal communication of 21).

fibre, forming permanent covalent bonds, raises the question of the effect of light on colorations produced by such chemical interaction. All the information available on the light fastness of the Procion dyes is summarised in Table V. The fastness is high in nearly all cases. Earlier work in this laboratory (Baxter, private communication) has shown that some other colorations formed by reaction with the fibre also are fast to light. Reactions of this nature might be expected to result in a type of chemisorbed monolayer of dye in the fibre which would have low light fastness, because normal dyes applied by methods favouring the formation of monolayers have very low light fastness 31. The high fastness obtained in the present cases suggests, therefore, that the reactions with the fibre are not as simple as might be supposed.

dyes against their relative depths, straight lines can be obtained which differ in slope from one dye class to another, probably on account of physical differences between the classes 24. These lines are referred to subsequently as characteristic fastnessgrade (CFG) curves, and statistical analysis (given in Appendix 2, pp. 157-158) shows that they may reasonably be considered as truly linear, and also that the differences between them are significant. This method of examining fastness data has now been extended by the use of a much larger range of dyes and fibres, so that more reliable comparisons can be made. Inferences drawn from the results can now also be compared with the results of laboratory fading experiments 7. These experiments show that there is a linear relationship between the logarithm of the time of fading  $t_F$  of a

TABLE V

	Light	Fast	ness	(B.S.	1006) 01	Procion	(ICI)	Dyes ****			
Procion dye		***		Yellow	R	Brilli	ant Re	d 2B		Blue 3G	1
Relative depth			4	1	03	4	1	2	5	1	2
Cotton, padded			6	6	6	5	5-6	5-6	6-7	6-7	-
Cotton, printed	***		-	6	-	-	6		-	6-7	-
Linen, padded			-	6-7	-	-	6	44-19	***************************************	7	
Linen (crease-resisted)	, padde	d	-	6-7	-		4		-	6-7	
Viscose rayon, padded			6-7	6-7	6-7	.5	5-6	6	6-7	6-7	_
Viscose rayon, printed				6-7	-	-	6 - 7	-	_	7	-
Nylon, dyed			5	6-7	6-7	4	5	5	.5	6	-
Silk, dyed			-	7	****		5	-	-	6	-
Silk, printed			-	6-7	-		-6	-		6-7	-
Wool (chlorinated), pri	nted		_	7	m-mil	-	6	-	-	6-7	

First there may be some form of penetration by the dyes of crystalline regions of the fibre. The reaction between dye and fibre must, of course, be more energetic than normal dyeing processes, so that it should be capable of breaking hydrogen bonds in the crystalline parts of the fibre, which normally resist the aqueous solutions used in dyeing. Dye combined in these regions will, after subsequent drying, be more firmly embedded and less accessible to air and moisture than dye in a normal fibre. Secondly, it is not inconceivable that some form of mixed dye-fibre aggregates may be present. Under wet conditions the molecular chains of the amorphous regions of the fibre are virtually in a state of solution and might readily become combined in new fibre-dye micellar structures\*. This would occur most readily with the least crystalline fibres, and in fact these do give better fastness in pale shades of these dyes than the more crystalline. Urea is added to the printing pastes, but it has no effect on the light fastness (cf. footnote on p. 139).

In the next section an attempt will be made to use all the available data to give more information about the physical state of the dyes in the fibres.

### IV—Rate of Increase of Fastness with Increase in Depth and its Dependence upon Physical State of Dye and Fibre

It has already been reported that, by plotting average light-fastness figures for whole classes of

given proportion of the original dye concentration  $C_0$  and the logarithm of  $C_0$ . The curves so obtained are called *characteristic fading (CF) curves* $\uparrow$ . The present CFG curves therefore differ from the CF curves only in using the fastness grade in place of the log  $t_{\rm F}$  value for the vertical axis. It is shown experimentally (see below) that fastness grades and log  $t_{\rm F}$  values can be considered equivalent, and therefore the CFG curves are assumed to be similar in nature to CF curves. The latter were shown, by theoretical and experimental investigation, to be capable of giving information on the physical state of the dye in the fibre, and the CFG curves should therefore do the same.

A detailed set of data on mean fastness grades of over 100 examples of dye-fibre systems is shown in Tables VI and VII, and a few typical CFG curves are given in Fig. 14 and 15. The list includes practically all the important types of dye and most of the commercially important fibres. The last column of the table gives the slope of the CFG curve, which represents the increase in average fastness grade for a tenfold increase in depth of dyeing. With a few exceptions (distinguished in the table) the average fastness figures are calculated only for dyeings having light-fastness grades between 1-2 and 6, inclusive, on the B.S. 1006 scale 52-54, and only dyes of which all depths have fastness between these limits are included. The reason for this is that the standards from 1 to 6 are evenly spaced 49, but standards 7 and 8 are less well spaced 45, and grade I assessments include lower degrees of fastness, for which

If so, this type of aggregation might occur also with ordinary dyes.
 Results of fading-rate tests made on Procion dyes in this laboratory (Catchpole, private communication) are consistent with the presence of aggregated material.

<sup>†</sup> This term is preferred to that formerly used.

Table VI .

Mean Light-fastness Values for Various Dye-Fibre Systems

		Mean Light-fasti	ness Value	s for Va	rious Dye	-Fibre S	Systems		
Dyes Class	Maker	Fibre	Ref- erence		of Dyes Selected		fean Fastne relative de		Slope† of CFG
						1	1	2	Curve
Basic—			WATER-	SOLUBLE	DYES				
Ordinary	BASF	Cotton (tannin mordant)	(5a)	38	38‡	1.03	1.06	1.50	(1-4)
Deorlene	Ciba	Orlon 42	(16r)	7	7	4-4	5-2		(1.7)
Direct-									
Ordinary	FBy	Cotton	(28e)	188	171	2.13	2.65	3:05	1.2
		Viscose rayon	(28e)	188	171	2-88	3.43	4.00	1.4 3
	CDC	Cotton	(17d)	88	49	2.74	3.01	3-41	(0.9)
	Gy	Cotton	(S9m)	116	68	3-15	3.62	4.00	1.05
	LBH	Cotton	(266)	75	46	2.3	3.0	3.4	1.7
		Viscose rayon	(346)	75	46	3.0	3.65	4.2	1.5
		Cotton	(35c)	9	9	2.5		3-1	0.8
		Wool	(80)	0	9	3.7		4.1	0.5
	ICI	Cotton	(a2f)	71	29	2-1	2.6	2.9	1-1
		Nylon	(831)	37	25	2.5	2.7	2.9	0.8
		Silk	(87A)	11	11	2.8	3.5	3-9	1.5
		Cotton	( <sup>87f</sup> )	71	116	1.80	2.35	2.85	1.3
		Viscose rayon	(879)	13	115	3.7	4-0	4.5	1.5
	В	Cotton	(50r)	121	71	2-65	3.32	3.93	1.6
	171141	Silk	(50a)	96	35	3-65	4-15	4.60	1.3
	YDC	Cotton	(636)	24	23	4-2	4-7	5-1	1.3
Levelling, for	ICI	Nylon	(871)	6	5	2-4	2-6	3.0	0.9
rayon	1713	Viscose rayon	(370)	24	22	3.3	3.9	4-4	1.4
Light-fast	FBy	Cotton	(20c)	59 59	33	4.50	4-80 5-23	4-91 5-65	1.0
	CDC	Viscose rayon Cotton	(174)	64	64‡	5-15	5-6	5-9	0.95
	CDC	Cotton	(178)	64	41	4.57	5.10	5-42	1.1
	Gy	Cotton	(104)	25	251	5-6	6-2	6.5	1.2
	LBH	Cotton	(866)	14	14:	4.7	5-2	5-7	1-3
	ICI	Cotton	(82)	40	38	3.80	4.55	4-96	1.5
		Nylon	(878)	9	8	3.9	4-2	4.4	0.7
		Silk	(87A)	11	11	3.9	4-4	5-0	(1-1)
	YDC	Cotton	(max)	24	241	5-7	6.2	6-5	0-85
	ICI	Cotton	(37)	40	214	3-4	4.05	4-4	1.3
		Viscose rayon	(270)	37	216	4.4	4.95	5.5	2.0
	8	Cotton	(50r)	73	44	4.68	5-20	5-50	1.2
Diazotised and	FBy	Cotton	(E85)	90	71	2.44	3-14	3-58	1.45 }
developed		Viscose rayon	(286)	90	71	3-16	3.90	4.35	1.55 \$
	ICI	Cotton	(27f)	29	15	2.8	3-2	3.5	0.9
Coupled with	FBy	Cotton	(E86)	35	29	2.14	2.69	3.19	1.4
diazotised base		Viscose rayon	(286)	35	29	2.52	3-21	3.91	(1.8)
Aftertreated with Cu or Cu + Cr	Ciba	Cotton	(160)	16	16	3.3	3-9	4-5	(1-6)
Aftertreated with Cu	8	Cotton	(500)	28	17	4.5	5.3	5-7	1.6
Ordinary +	ICI	Nylon	(871)	111	128	3.4	3.7	4-2	(1.0) 5
light-fast		Cotton	(27)	111	125	3.0	3.5	3.9	1.1
classes combined		Viscose rayon	(379)	111	128	3.9	4.3	4-16	1.2
Water-soluble sulphur	ICI	Cotton	(3799)	10	10	3-4	3-75	4.05	0.9
Water-soluble sulphur, aftertreated with Cu and Cr	ICI	Cotton	(2700)	5	5	4-5	5-0	5-2	(0-95)
Wool—									
Levelling acid									
(Acilan)	FBy	Wool	(and)	133	70	3-54	4-11	4.55	1.3
(Supramine)	FBy	Wool	(50d)	23	18	4.05	4:6	5.15	(1-4)
(Supracen)	FBy	Wool	(20d)	15	10	5-2	5-6	5.9	0.9
	CDC	Wool	(174)	40	34	3-75	4.36	4.87	1-4
	Gy	Wool	(20c)	72	39	2.55	2-94	3.67	(1.4)

10		1211-00		VI— con	of Dyes	Mon	n Fastness*		Slopef
Class	Maker	Fibre	Ref- erence		Selected		lative depth		of CFG
						1	1	2	Curve
Wool—continued- Light-fast		Wool	(86r)	63	38 .	4.21	4-74	5-28	(1-1)
rugue-mae	Gy		(871)	65	46	3.87	4.07	4.40	0.6
	ICI	Nylon Wool	(974)	73	51	3.50	3.95	4.49	1.5
	CI.		(846)	150	104	3.55	4.07	4-62	(1.4)
	8	Wool Silk	(50a)	27	15	3-95	4.55	4.85	1.2
	YDC	Wool	(890)	59	53	3.7	4.4	4.9	1.8
MATERIA	100	** 001	5 /	00	-		-		
Milling acid—	1210	W1	(284)	40	35	3-58	4.21	4-67	1.4
(Supranol)	FBy	Wool	(174)	46 36	28	3.80	4.35	4-85	1.3
(Benzyl Fast) (Erio Fast S)	Gy	Wool	(30e)	19	16	4.25	4.85	5-35	1.3
(weak acid	Gy	Wool	(30e)	58	37	2.76	3.54	4.09	1.95
(Polar)	Gy	Nylon	(20d)	27	21	3.6	4.5	5-2	2.0
		Wool	(30e)	31	21	4.2	4.7	5-3	(1.5)
(Carbolan)	ICI	Nylon	(271)	12	11	5-2	5-2	5-3	(0-35)
		Silk	(37h)	12	11	4-4	4.9	5-2	0.95
		Wool	(374)	12	11	4-7	5-4	6.0	1.6
(Coomassie)	ICI	Nylon	(878)	31	21	3.2	3.5	3.8	0.8
		Wool	(374)	31	21	3-9	4.5	5.05	1.5
	8	Silk	(50a)	65	47	3-8	4-4	5.0	(1.5)
		Wool	(506)	97	70	3-74	4.26	4.78	(1-4)
Acid milling	8	Silk	(50a)	65	29 §	3.5	4.2	4-6	1.4
		Wool	(606)	65	29	3-8	4-4	4.8	1.3
	YDC	Wool	(534)	37	35	3.3	3-9	4.5	(1 - 2)
Neutral-dyeing	Gy	Wool	(390)	20	8	2.8	3.3	3.9	(1.8)
Levelling +	8	Silk	(50e, 5)	75	58	3-40	4.08	4.46	1.5
milling classes combined		Wool	(*************************************	75	58	3.83	4-32	4-79	1.6
Chrome—									
Afterchromed	FBy	Wool	(38*)	136	39	3-82	4.65	4-95	(1.5)
				136	109‡	4-62	5-21	5-74	1:4
	CDC	Wool	(178)	64	27	4-10	4.67	5.21	1.4
Afterchromed	LBH	Wool	(26e)	44	37	4-6	5.3	5-9	1.7
and chromate	Gy	Wool	(200)	117	57	3.83	4.40	5-08	(1.5)
					88‡	4-44	5.07	5.73	(1.6)
	ICI	Nylon	(874)	43	25	4.8	5-2	5-6	0.85
		Wool	(37k)	39	23	4-1	4.6	5-3	(1.2)
Chromate	FBy	Wool	(904)	44	20	3-8	4-4	5-1	(1.6)
process	$_{\mathrm{Br}}$	Wool	(13)	19	18	4-1	4.8	5-4	1.7
Metal-complex-									
Acid-dyeing (Ultralan)	ICI	Silk	(87A)	12	11	3-7	4-1	4.6	1.1
Neutral-dyeing (Cibalan) (Lanasyn)			. 403	49.49	044	0.0	0.5	0.7	0.05 3
	Ciba	Nylon	(¶)	26	241	6.0	6-5	6-7 7-0	0.95
	-	Wool	(196) (And)	26	24‡	6.0	6.6	7.3	1.6
	8	Nylon	(bod) (bod)	14	14;	6.0	6-6	7-1	1.4
	-	Wool	(100)	14	14#	5.5	6-1	6-6	1.4
(Irgalan)	Gy	Nylon	(000)	14	11‡	6.0	6-6	6.9	1.2
		Wool		PERSE D		0.0	0.0	0.0	
	BASE	Cellulose	(55)	53	26	4-3	5.0	5.5	1.6
		acetate**	(56)	53	125	4.7	5.3	5.7	1.3
		Orlon	(8e)	12	125	6.3 (412 11)	6-7 (1)	6.9 (1)	(1.0)
	$\operatorname{BrC}$	Cellulose acetate**	(13)	32	29	4-8	5-4	5-8	1.3
	Ciba	Bright cellulose acetate**	(10a)	35	21	3-9	4.7	5-6	(1.9)
			(160)	35	21	4.1	4-75		1.3
		Dull nylon Bright	(104)	35	5	4-1	5-0	Record	1.8
		cellulose	( )	1212	911				
		acetate**							1
		Bright cellulose	(10)	35	5	3.6	3.9	-	0.7

			TABLE	VI-con	tinued					
Class	yes Water	Fibre	Ref-		of Dyes		Mean Fastne		Slop	
Class	Maker		erence	Total	Selected		t relative de		of CI	
	G.	D-1-14	/90as	0.0	10	1	1	2	Cur	70
	Gy	Bright cellulose acetate	(200)	22	19	4.5	5-4	5.9	1.9	1
		Dull nylon	(204)	22	190	3-4	4.3		1.0	1
	LBH	Bright	(264)	42	38	4-1	4.7		1.9	3
	Linit	cellulose acetate**	(-)	9.0	38	4.1	4.7	5.2	1-4	
		Dull nylon	(80)	40	33	4.0	-	4-7	0.9	
	ICI	Bright cellulose	(271)	49	22	4.3	4.8	5.2	1.2	1
		Dull cellulose	(876)	49	22	4.25	4.7	4.9	0.9	}
		acetate**								
		Dull nylon	(871)	27	221	3.5	3.8	4.0	0.7	j
		Bright cellulose scetate**	(376)	19	19	4:4	5.0	-	1-3	1
		Bright	(8786)	19	19	4.2	5.4		2.5	1
		Courpleta Dull nylon	(371)	27	23	3.6	3.9	4.2	0.74	3
		Terylene	(371)	27	23	3.8	3.9		0.75	36
	YDC	Cellulose	(60r)	42	20	4-4	4-9	4·0 5·3	1.2	3
	120	acetate**								1
		Cellulose triacetate	( <sup>end</sup> )	53	20	4-2	5.0	-	1-9	1
		Nylon Cellulose	(68e)	36	20	3.6	4.4	4.7	1.4	1
		acetate** (bright)		5	5	5-8	6.3	6-6	1.0	1
		(pigment- delustred)	( <sup>60r</sup> )	5	5	5.3	5-8	6-1	1.0	Ì
		Terylene (100°C., no carrier)	(eq)	15	7	(4.6)?	4.6	(4-6)?	св. 0	
		(dyed at high temp.)	( <sup>68A</sup> )	15	7	5.4 (2%)	5.7 (7.5%)	-	0.65	
(cf. T	able XIV)	Bright cellulose acetate**	(Table XIV)	32	13	5.0	5.6		1.3	1
		Bright cellulose triacetate	(Table XIV)	32	13	3.5	4-3	-	1.7	)
		Bright cellulose triacetate	(10)	47	42	3-85	4.45		1.2	
		Dyes	INSOLUB	ILISED D	THE F	BRE				
Azoic-	Various	Cellulose acetate**	(14)	5	5‡‡	2.5	2.6	3.4	(1-17	17
		Viscose rayon	(14)	5	5	3.7	4-8+	5.6+	2.4+	)
	CDC	Cotton	(17c)	44	23	3.35	4.54	5.46	2-7	
				44	44:	4.05	5.30	6-15	2 2	
	ICI	Cotton	(37c)	294	202	4.06	4.56 (111)	5-16 (1††)	2-4	
				294	2941	4.52	5.07 (111)	5-85 (1††)	2.7	
Sulphur	LBH	Cotton	(36e)	23	23	3.1	3.9	4-8	$(2 \cdot 3)$	
Aftertreated v		Cotton Cotton	( <sup>40</sup> ) ( <sup>60</sup> )	148 148	129 129	2·27 3·97	3·36 5·13	4·25 5·69	3-3 (2-8)	
Cu and Cr	DAGE	C-H	(54)	62	204	0.0-	20.00			
Vat—		Cotton	(58)		57‡	6.25	6-60	7.05	1-0	
	Gy	Cotton	(88r) (866)	58	29	4-58	5-18	5-78	(1.5)	
	1.011	Cotton		92	921	5-5	6-15	6-66	1.5	
		Cotton	(35d) (37d)	51 86	50\$	6-1	6.7	7-1	1-3	
	ICI	Cotton	(874)	86	18 741	3.6	4.5	5.0	1.8	
Solubilised (indigoid)	ICI	Cotton	( <sup>37m</sup> )	8	8	5.73 2.5 ( ††)	6·28 3·2 ( <u>1</u> ††)	6-65 3-8 (1††)	1-3	
(anthra- quinone)			(87m)	16	16‡	5-4 ([††)	5-9 (111)	6.2 (1††)	0.9	
Indigoid (applied fro	ICI	Cotton	(374)	8	5	3.0	4-1	4.7	2.1	7
vat)	***		(97m)							1
(applied solubilised)			()	8	5	2-7 (111)	3-4 (1/1)	3-9 (1††)	1.2	1

			TABLE	VI-con	tinued				
Class Dyes	Maker	Fibre	Ref-	No. o	of Dyes Selected		ean Fastnes relative der		Slope†
						å	1	2	Curve
Vat - continued -									
Anthraquinone (applied from vat)	ICI	Cotton	$(^{37d})$	16	10	6.7	7.0	7.3	0.8
(applied solubilised)			(37m)	16	10	5-8 (111)	6-3 (111)	6-5 (1††)	0.7
		Viscose rayon	(37m)	16	16‡	6.5 (111)	6.7 (111)	7.0 (1††)	0.5
Anthraquinone	ICI	Viscose rayon	(570)	70	651	6.40	6-90	7-40	(1.3)
Indigoid				10	10	4-4	5.0	5.75	(1-7)
			1	GMENT	4				
	BASF	Viscose rayon	(50)	20	181	6.7	6.95	7-1	0.5

· All intermediate grades calculated as half-units.

† The slope is the increase in average fastness grading for a tenfold increase in depth. Slope values in brackets (\(\frac{1}{2}\)\)) are those where the three points do not lie on a straight line. Those in braces (\(\frac{1}{2}\)\)) are for identical selections of dyes on the two fibres concerned.

Selections including many fastness grades outside the limits of 1-2 to 6 (see above). § Series selected from larger classes given elsewhere in the table for particular comparisons between two fibres.

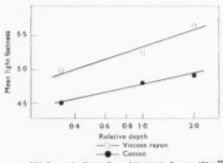
|| A few 6-7 values included to complete the series.

¶ Private communication.

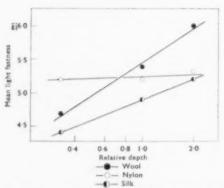
. Secondary cellulose acetate.

†† Relative depths.

:The depths on the two fibres are stated not to have been identical.



(a)— CFG Curves for Direct Dyes of High Light Fastness (FBy) <sup>887</sup> on Cellulosic Fibres of Different Regain



(b)—CFG Curves for Milling Acid (Carbolan) (ICI) Dyes 27a, h, f on Fibres of Different Regain

Fig. 14-CFG Curves for Fibres of Different Regain

no standard is available. To make Table VI truly representative of all commercial dyes, however, data for basic dyes, which include many grade 1 figures, are given, and also several series of data for dye classes of the highest light fastness, with many fastness figures higher than 6. These series are suitably differentiated in Tables VI and VII. Standards 7 and 8 are said to be more widely spaced than the others 45, so that the slopes obtained by

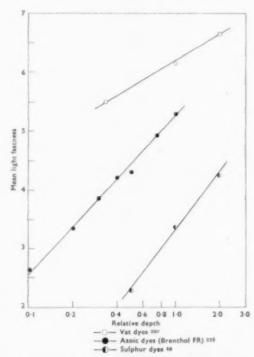


Fig. 15—CFG Curves for Dyes insolubilised in the Fibre (on Cotton)

plotting curves based on these grades will be lower than the true values.

The distribution of slopes over most of the main classes of dyes and fibres is shown graphically in Fig. 16, from which it is evident that there is in many cases a marked tendency for slopes to differ between dye classes or between fibres. The differences must represent real differences between average properties of the respective dye-fibre systems. The work on CF curves <sup>7</sup> showed that their slopes probably reflect differences in the particle-size distribution of dye in the fibre. It is therefore assumed that the slopes of the CFG

TABLE VII

	Slope		3.0	(9.8)	0.0	3.0	10.4
		*(0)	1	(5-05)		(5-51)	(5.30)
		*(1)	(2.06)	4.37	4.92	4.70	4.94
	2	(e)	4.34	28.83	4.10	4-19	4.30
	lean Light Fastness	(p)	4.30	3.00	3.80	4.03	4.21
	an Ligh	(c)	3.76	3.13	3.53	3.60	3.85
0	Me	(9)	3.19	2.30	3.04	2.94	3.34
n Cotto		(0)	(2.43)	(2.38)	(2.30)	(2.52)	(5.64)
ight-fastness Gradings for Azolc Dyes on Cotto		(0)	1	1-35	1	1.75	1.0
		S	1.25	0.95	1.35	1.0	0.75
adings	bre (%)	(e)	0.85	19-0	8.0	0.73	0-2
ness Gr	ol on Fi	(p)	0.72	0+40	0.64	0.63	1-0
ght-fast	Brenth	(0)	9.0	0.34	0.47	0.3	0.3
fean Li		(9)	0-4	0.55	0.25	0.3	0.5
·		(a)	0.5	0-125	0.13	0.15	0.1
	No. of Bases	24	52	000	0.00	24	
	coupling Component of Arylide of 3:2-Hvdroxv.	naphthoic Acid 36	a-Naphthylamide	Anilide	4-Bromo-2-methoxyanilide	5-Chloro-2-methylanilide	o-Anisidide
	Brenth (ICI)		AN	AS	BA	5	FR

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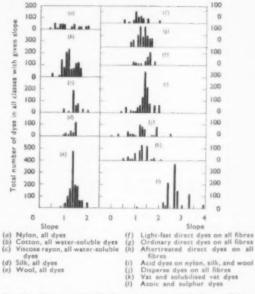
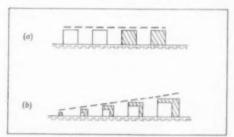


Fig. 16 — Frequency Distributions of Slopes of CFG Curves (Tables VI and VII) arranged by Fibre Class and Dye Class (all manufacturers.

curves are similarly related to particle size. To enable the reader more readily to follow the interpretation of the slope differences, a summary of some of the conclusions reached earlier is now given.

#### SIGNIFICANCE OF SLOPE VALUES

As already mentioned, the rate of fading depends upon the surface which the dye presents to light and air. A given concentration of dye will fade more slowly in large particles than in small, because of the smaller exposed surface. In any dyed fibre there may be a wide range of sizes of dye particle, varying from molecularly dispersed or monolayer material to aggregates of considerable size (which in the case of some insolubilised (azoic) dyes are actually visible under the microscope 8). The slope of the CF or CFG curve is considered to be related to the rate at which the distribution of sizes of the particles changes with increase in depth of dyeing. This can be illustrated simply as follows—



increase only in number of particles

(b) Increase in range of sixes of particles reas Original amount of dye White areas

Hatched areas Dye added to double concentration

Fig. 17 — Schematic Illustration of Two Modes of Particle Growth with Increase in Dye Concentration in a Fibre

Fig. 17 shows two hypothetical systems of dye particles, set in a channel between the molecular chains of a fibre, and suggests possible ways in which they may alter in size and shape when more dye is added.

System A (cf. Baxter et al.7, Fig. 1A) consists of dye particles which are large and uniform in dimensions. They have little tendency to increase in size, and if the dye concentration is increased additional particles are formed, having similar dimensions to the original ones. There is therefore little or no difference in the range of particle sizes at various concentrations. This type of system should give a CFG curve of low slope. (Theoretically, if the range of sizes remains constant, the curve should be horizontal, but this never seems to occur with soluble dyes.) A low slope arising from uniformity of particle size could conceivably occur under two opposite conditions— (a) when the dye forms only very small particles, or even a monolayer; and (b) when the dye forms very large particles, which tend to be uniform in size because of restriction of their growth by the internal structure of the fibre. Fading would be expected to be rapid in case (a) and slow in (b).

System B (cf. Baxter et al.<sup>7</sup>, Fig. 1B) consists of dye particles of different sizes, and when the dye concentration is increased these may grow larger and also other small particles may form, resulting in a still wider range of sizes. This type of system should give a CFG curve of high slope.

As a simple aid to memorising this suggested connection between the nature of the particles and the CFG slope, lines may be drawn as shown (Fig. 17), just touching the tops of each particle in the diagrams. The slopes of these lines follow the same order as those of the corresponding CFG curves: A has a low, and B a high slope; but they have no significance other than as a mnemonic device.

On the basis of the above hypothesis certain predictions concerning light fastness can be made and tested by an examination of the present data. These predictions are as follows—

(a) High light fastness should be associated with both low and high slope values, and low light fastness with intermediate slopes

(b) The slope should generally be higher the higher the regain of the fibre

(c) Dyes insolubilised in the fibre should tend to give high slopes

(d) Colouring matters applied as pigments should give very low slopes.

The reasons for these predictions, and the extent to which they agree with the present results, are now discussed.

#### (a) Relationship between Slope and Maximum Fastness

It is conceivable that high light fastness in deep colours could be associated with either of two types of system—type A (see above), in which the dye has a natural tendency to form uniformly large aggregates, or type B, containing a high proportion of large aggregates. The former system will have a

low, and the latter a high, CFG slope. It follows, then, that slopes of intermediate value may be associated with lower light fastness. (Dyes present predominantly as monolayers or very small particles should also have very low light fastness and give curves of low slope, but it is believed that very few dyes fulfil this condition cf. 7. 31.)

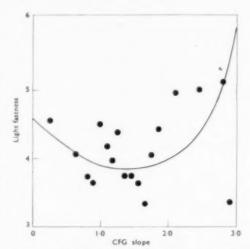


Fig. 18—Relationship between Light Fastness of Medium Depths and Weighted Mean Slope of CFG Curves (Best parabola fitted by method of least equares) (Data from Table VI; classes with gradings up to 6 only)

This hypothesis was examined thus: weighted mean values\* for fastness in medium depths were plotted against slopes (Fig. 18), and the best parabola was fitted to the points. For convenience, all the slopes were divided into a series of short ranges, which are shown in the graph by a point representing the middle of each range. The data embodied in this curve were obtained from over 12,000 measurements and should give significant information. There is in fact a marked tendency for high light fastness to be associated with both low and high slopes, and for low fastness to be associated with intermediate slopes, in accordance with the prediction.

#### (b) Relationship between Slope and Regain

The larger the maximum pore size in the fibre, the larger should dye particles be able to grow, and hence the wider should be the distribution of their sizes. Thus in a comparison of identical series of dyes on pairs of fibres of different regains, the rule should be that the slope of the CFG curve is greater on the one of higher regain. All the possible comparisons of dye classes on pairs of fibres are set out in Table VIII, from which it will be seen that out of 41 no less than 29 follow this rule†.

The twelve anomalous pairs can be divided into two classes, in which the slope is higher than expected in (a) one series of dyes on each of four fibres of "normal" regain, viz. cellulose acetate (dull), cotton, silk, and viscose rayon; and (b)

i.e. Means calculated by first multiplying the individual slope values by the number of dyes they represent.

<sup>†</sup> These figures include comparisons with pigment-dejustred cellulose acetate, assumed to have lower porosity than the bright fibre.

TABLE VIII

#### Comparisons of Slopes of CFG Curves of the Same Dye Classes on Fibres of Different Regain

00	mplan 10	OHO U	i Giope	01 01 0	Cur ves or	the built					
	Fi	ibre					Dye Man	ufacture	rs er		
				FBy		LBH		ICI		YDC	
					DIRECT	Dyes, O	RDINARY				
	Nylon							0.8	-		
	Cotton		4.00	1.2	}	1.7		1.3	}	0.8	
	Viscose		)B	1.4	)	1.5		1.5	)	0.5	
	Wool		4.0.0			-				0.00	
					DIRECT	DYES, LI	GHT-FAST				
	Nylon					_		0.7			
	Cotton			0.5	)	1.3		1-3	1	0.85	
	Viscose			1.0	}			2.0	3		
					DIRECT I	DYES, WHO	DLE RANG				
	Nylon		* * *					(1.0)	)		
	Cotton							1:1:	7		
	Viscose	e rayo	n			-		1.2	1		
				DIRECT I	YES, AFTE	RTREATED	(VARIOUS	PROCES	ses)		
	Cotton		***		45 \		( · mmoco		,		
	Viscose			1.8				-		_	
	* 2000,000	· ing	,,,,,,		,						
				CDC		Gy		ICI		8	
				CIN	Acm	LEVELLIN	DYES.	20.2			
	Nylon			4	25.10			0.6			
	Silk			-		#E70-0				1.2	
	Wool	***		-		-		1.5		(1-4)	
	*** 001		***							1,	
					Acm	MILLING					
	Nylon			_		2.0		0.35 7 0	.8 7	-	
	Silk			-		- }		0.95	- }	14 }	
	Wool	***	***	*****		(1·5) J		1.6 ] 1	·5 J	1.3	
					Acm D	YES, BOTH	CLASSES				
	Silk		***		240.100 22					1.5	
	Wool	***				-				1-6	
	******	***	***								
					, Chromi	E (AFTERC	HROMED)				
	Nylon			_		Birmed.		W-1000		0.85	
	Wool	4.4.4	4.4.0	-		*******				(1.2)	
						3.6					
	NT. T				NEUTRAL-D		TAL-COMP	LEX		10 5	
	Nylon			0.95	}	1.4				1.6	
	Wool			1.3	,	1.2 )		-		1-4	
			DAG	D (	liba	Con	LBH		ICI	VIVI	Various
			BAS			Gy ISPERSE D			101	YDC	Various
0.1			71.01	1	120	INPERNE D	E ESS				
Orlon	***	***	(1.0)						0.05.	0.000	
Terylene	2.4.3	4.47	-				_		0-25	0-65*	
Mades (dol	115				1.9 %	1-9 7	0.9	0.5	0.75	*	
Nylon (dul		44.0	_	0.7	1.3			0-3		1-4	1.7
Cellulose to (bright)	racetat	B		0.7	1 -}	- }	A-1 -120		2.5	1.9	1.7
Secondary	asllulas	Left.	1.3	1.8	1.9	1.9	1.4	10	11.3	1.2 1.07	1.3
acetate (			1.0	1.0	1.02	1.0 -	1.4	117	1.0 )	1.22 1.0	1.0 )
(dull)			-				*-000	0-1	,)	- 1.05	
				)							
						Azoic Dyi	80				
Secondary											
acetate (			-								(1-1) }
Viscone ray	/on		emment		-	B	_		***	44.000	2-4+ 5
						VAT DYES	t.		4		
Cotton			-		entered to the same	****			1.2		
Viscose ray					-	g-10-0			(1.3)		
									(2.07)		

<sup>•</sup> Dyed at 125°c.

t Very low (dyed at 160°c). (No figures given for pale and heavy dyeings, but fastness stated to be virtually the same at all depths.)

The solubilised vat dves give a lower slope on viscose rayon than on cotton (Table VI), but this is undoubtedly due to far more dves having gradings above 6-7 on cotton, grades at this level being more widely spaced (cf. p. 140).

eight series on low-regain fibres, viz. five on nylon and three on cellulose triacetate.

It seems reasonable to assume that the four anomalies under (a) are random effects probably caused by differences in the atmospheric conditions prevailing during testing, but that the eight under (b) are systematic, and have their origin in some property of the dye-fibre system. All these eight series include dyes which are likely to form a high proportion of large aggregates and the two fibres which have regains (cf. Table II) at about the level at which the diffusion-restriction effect begins to be apparent. If this effect does occur with these fibres it would be expected to be more noticeable at high than at low dye concentrations, because of more complete blockage of the fibre pores; the result of this would be a CFG curve of higher slope than normal, as is found in the present anomalous

The tendency for the slope to increase with fibre regain for a given series of dyes on nylon, cotton, and viscose rayon can be seen in a more general way from the distribution graphs of Fig. 16. Wool and silk do not show the effect, but the number of dyes examined on silk is probably insufficient for a significant result.

#### (c) High Slopes of Insoluble Dyes

The intermolecular forces which cause the dye molecules to associate will be more powerful if the dye has no attraction for water and consequently is not protected by an atmosphere of solvated water molecules. It is therefore to be expected that the dyes which are insolubilised in the fibre, usually by a rather rapid process of precipitation from solution, will form larger particles in a wide range of sizes, and therefore will show higher CFG slopes than those which remain water-soluble after dyeing.

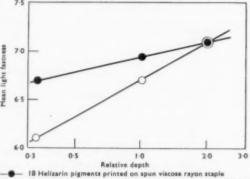
Fig. 16 shows that the distribution of slopes is actually much more in favour of very high values for the insoluble (azoic and sulphur) than for the soluble dye classes. (The theoretical upper limit of slope in normal fibres is about 4-65 in the present units 7, and some of the azoic dyes approach this.) The vat dye classes do not give high slopes. This must be due partly to their high proportion of grades above 6 (see above, p. 144) and partly to the uniformity and small size of their particles. They are known to form smaller aggregates than the azoic dyes 8,56.

#### (d) Low Slopes of CFG Curves for Pigments

According to the argument of the previous paper 7 any colouring matter applied in different concentrations as a pigment, so that it is not in solution at any stage of the process, should, because of its uniform particle size at all concentrations, give a horizontal CFG curve: i.e. the light fastness should be the same at all concentrations, and should not fall with decrease in strength, as it does normally when dyes are applied from a solution. Thus to achieve the highest possible fastness in pale colours the application of a pigment is theoretically preferable to a dyeing process. Certain interfering factors, however, may tend to prevent complete

constancy of fastness of a pigment over a range of concentrations.

In the first place, adjacent particles may screen each other from moisture, etc. to an increasing extent as the concentration rises; this might occur particularly if they are not perfectly evenly spread, which, in fact, they are very unlikely to be, clumps probably being present. Thus a pigment dispersion may be effectively more aggregated at higher concentrations than at low, even though the original particles remain the same size. An analogous case will be found in the process of colour photography by the mosaic-screen process, where a screen consisting of a printed réseau of colour rectangles is found to give a finer resolution in the image than one consisting of a random mixture of particles, even though these are individually finer than the rectangles of the réseau; the reason for this is that the particles form many irregular clumps, according to the laws of probability 18. Secondly, differential migration effects may occur during drying, particularly in films. Thus pigments dispersed in gelatin films give CF curves with flatter slopes than those in Cellofas A, because the former sets more rapidly and so hinders migration (Baxter et al.7, Fig. 4).



18 Indanthren vat dyes applied to cotton from the vat (selection of these dyes having identical fastness to the Helizarin dyes in deep colours)\*

Fig. 19—CFG Curves for Water-insoluble Dyes applied as Insoluble (Pigment) and temporarily Solubilised Forms\*

• To make this comparison as true as possible, the vat dyes were selected to have the same mean grading in deep colours as the Helizarin pigments. They comprise Indanthren— Yellowa 76K and 46F; Oranges RETS, FSR, and 4E; Reds GG and FBB; Brilliant Pink BBL; Rubines GR and B; Bordeaux B; Turquoise Blue 36K; Brilliant Greens B, FFB, and GG; Maroon BR; and Grey RERL 55-6.

Nevertheless, pigments applied to fibres as dispersions do appear to give flatter CFG curves than dyes of comparable fastness applied as solutions and insolubilised in the fibre. Thus Fig. 19 shows that the Helizarin pigments give a flatter CFG curve than the most nearly comparable set of vat dyes of the same manufacturer.

It should also be mentioned that pigments used in paints are often much less fast to light in pale than in deep colours <sup>37x</sup>; this is perhaps attributable to the influence of other photochemically reactive ingredients (e.g. extenders) in the film, which are necessarily present in relatively increasing quantities with decreasing pigment strength.

#### SLOPES OF DYES ON NYLON AND CELLULOSE TRIACETATE

The moisture-diffusion effect in low-regain fibres, referred to earlier (p. 137), must affect CFG slopes, and the effect is likely to be greater with fibres of intermediate than with those of very low regain. In the latter, e.g. Terylene, the pores are so small that they may be blocked, and consequently fading retarded, over the whole range of concentrations, and therefore, though the fastness may be improved, the CFG slope may not be greatly affected. Fibres which are on the borderline, having a regain at which the diffusionrestriction effect begins to become significant, e.g. nylon, cellulose triacetate, and perhaps even secondary cellulose acetate, may well behave differently. In these fibres the restriction may operate more at high than at low dye concentrations, and in consequence the slope may tend to be higher than expected. As discussed above, a comparison of slopes of dye classes on many pairs of fibres (Table VIII) does indeed show that nearly all the anomalies occur in cases where nylon or cellulose triacetate appears to give an abnormally high slope.

#### SLOPES OF DIRECT DYES

Some of the direct dyes of specially high light fastness are premetallised complexes of copper or other metals. These may owe their improved fastness largely to the protection of the centre of photochemical attack (the azo group) by chelation. Others, which contain no chelating metal, probably owe it to their improved physical state resulting from a tendency to form uniformly large aggregates. This latter appears to be the interpretation of their frequently low slopes (CFG curves) and the adverse effect of the crease-resist process.

#### CASES OF REDUCED FASTNESS WITH INCREASED DYE CONCENTRATION

Among all the data examined in this survey less than a dozen dyes exhibit the unusual phenomenon of a lower light fastness in deep than in pale dyeings. Two of these cases are disperse dyes on cellulose acetate 374, and the others are disperse dyes on Terylene 37t (without carrier). It would be hard to account for this effect if the dyes were molecularly dispersed, but a reasonable explanation is possible on the supposition that the dyes are present as aggregates, as follows. The growth of aggregates must cause considerable disturbance of the arrangement of molecular chains of the fibre. In some special circumstances this may so distort these chains that they are forced apart, forming gaps through which air and moisture can penetrate more readily than in the undyed fibre. This condition would be more likely to occur with high dye concentrations than with low, and if it does, then the greater ease of penetration of moisture would produce a more rapid fading at the higher concentration. The special conditions producing this effect are most likely to arise in a fibre with very limited amorphous regions, and it may therefore be significant that the effect is almost entirely confined to Terylene, which is the most crystalline of the fibres examined.

#### V-General Conclusions and Suggestions

On account of the great variety of factors which potentially may determine light fastness, no general rules can be formulated which will hold in all cases. This, of course, is the reason for the need to use statistical methods in examining practical data. The principles which emerge from this work must obviously, therefore, be subject to many exceptions, but they do appear to establish an integrated theory applying to the bulk of light-fastness assessments.

The present survey has confirmed the general applicability to all dyed fibres of the theories previously advanced, and also has revealed the operation of some factors not previously identified. Broadly, the following conclusions may be stated to follow from the survey—

#### 1. PHYSICAL STATE OF DYES

In dyed fibres the dye usually exists in particles ranging in size from single molecules to aggregates of various sizes\*. The proportion of each depends upon the nature of the dye and upon the internal physical form of the fibre: the more crystalline the fibre, the smaller the proportion of aggregates. Insoluble dyes, of course, must always be present as discrete particles, and it seems reasonable to assume that dyes which have low solubility or are colloidal (e.g. acid milling and disperse dyes) may associate in the fibre. It seems at first sight less likely for dyes which are mainly unaggregated, e.g. some acid wool dyes with small highly sulphonated molecules, to associate, though such dyes do appear to aggregate in some circumstances when insolubilised in monolayers on water (Cameron, private communication). Whatever may be their actual state in the fibre, however, it cannot be that of a completely accessible monolayer, for if it were, their light fastness should be the same at all moderate depths (see p. 146)cf. 7, whereas in most cases this is not so. The only other reasonable explanation of this fact appears to be that these dyes are present as a monolayer, but a proportion of their molecules become "trapped" between the molecular chains of the fibre and thus are rendered inaccessible to air, etc. cf. 7, the proportion increasing with depth of dyeing. This hypothesis has been tested by examining the fastness of dyes which are highly surface-active and therefore especially likely to form monolayers. On what may be called the "trapping hypothesis" the fastness of such dyes should not differ from that of normally unaggregated dyes, whereas in fact in many cases it differs considerably, their rate of fading being very high and changing less than usual with increase in depth (Baxter, private communication),

#### 2. PHYSICAL DETERMINING FACTORS

The principal factors determining the light fastness of any dyed fibre are physical, viz. the state of aggregation of dye and the ease with which

<sup>•</sup> A highly condensed monolayer might be expected to act as an aggregate, if the approach of water or oxygen to the reactive centre of the dye is retarded by the closes side-by-side packing of the dye molecules. There is, however, no evidence that this is so; in fact, the dyes which most readily form condensed monolayers often have very low light fastness \$1.35.

moisture (and oxygen) can diffuse towards the irradiated dye. Large aggregates fade more slowly than small ones, so that usually dyes are more resistant to light on the more porous fibres. Retardation of diffusion of moisture caused by blockage of the pores of the fibre by dye appears to become significant in fibres of very low regain and causes the degree of fibre crystallinity to influence fastness in the opposite sense to that observed on the more porous fibres.

#### 3. ORDER OF FASTNESS IN FIBRES

On the basis of these principles it is possible to arrange fibres roughly in the order in which they should affect the fastness of a hypothetical dye, substantive to all fibres. The order of fibres causing decreasing light fastness of such a dye would be approximately: wool, jute, linen, viscose rayon, silk, mercerised cotton, cotton, Terylene, Orlon, cellulose acetate, nylon, cellulose triacetate. The sequence is, however, likely to vary in practice, according to the nature of the dye tested.

#### 4. INCREASE IN FASTNESS WITH CONCENTRATION

The rate of increase of light fastness with dye concentration depends on the physical factors already mentioned, just as does the fastness at any given concentration. It rises with increase in heterogeneity of dye particle size, and in a few fibres it is further increased by restriction of diffusion of moisture. It is more likely to be high with dyes which are insolubilised in the fibre after dyeing than with those which are not changed. The average increase in fastness varies considerably from one dye class to another, but tends to be about 1½ units for a tenfold increase in dye concentration with soluble dyes and about 2½ units for insoluble ones.

#### 5. EFFECTS OF CHEMICAL REACTIVITY

The chemical reactivity of a dye plays only a secondary rôle in determining its light fastness. The difference between the chemical nature of fading on protein and non-protein fibres has a noticeable but slight effect, principally at the two ends of the scale. Thus dyes which have very low or very high fastness on cellulosic fibres are likely to have a grading about 1 or 2 units higher or lower respectively on wool. Other instances in which chemical reactivity influences fastness are (i) the abnormally rapid fading on nylon of some disperse dyes, which are active in tendering the fibre, and (ii) fading on crease-resisted cellulosic fibres. In the latter case, the small residual amount of formaldehyde apparently acts in some cases as a retarding agent in fading, particularly with the direct dyes of low fastness.

#### 6. CORRELATION OF FASTNESS AND CONSTITUTION

The comparative unimportance of chemical reactivity explains why attempts to correlate light fastness and dye constitution are usually unsuccessful, except in series of dyes differing only slightly from each other in structure. It also follows that no dye can strictly be said to have any particular light fastness. A fastness figure given to it applies only to one depth on a specified fibre.

Usually, of course, the quoted figure refers to a medium depth on the fibre for which the dye is normally used. Stead <sup>55</sup> has pointed out that no absolute value of light fastness can be given for a pigment, for a similar reason.

If all dyes were to exist in the same physical form in fibres, then their light fastness (on nonproteins or on proteins respectively) would probably run parallel to their resistance to chemical oxidation or reduction. Also, if there is a tendency for the physical form of all the dyes in one class to be similar on any given fibre, which does appear to be the case, then in that class of dyes high chemical stability will tend to be associated with good light fastness. This must account for much of the increase in fastness of dyes aftertreated with chromium or copper, because in the resultant dye-metal complexes the reactive centre of the dye (the chromophoric group) is protected against attack by inclusion in the chelate ring formed with the metal atom.

Aftertreatments do not noticeably improve light fastness in cases where they do not give protection to the active centre, e.g. the chroming of salicylic-acid azo mordant dyes (Baxter, private communication) and the diazotisation and development of direct dyes on the fibre.

#### 7. MEANS OF ACHIEVING HIGH FASTNESS

The highest light fastness seems likely to be found in dyes showing a marked tendency to intermolecular association, and these are likely to have low solubility in water or to have large planar molecules without strongly ionic groups at the periphery. Examples of such structures in actual use are the anthraquinone vat dyes and the phthalocyanines, both of which exhibit very good light fastness on most fibres. Soluble dyes with markedly non-planar molecules may have low fastness. This may account for the low rating of triphenylmethane derivatives. The simple benzidine disazo direct dyes usually have low fastness on cellulose, probably because of their tendency to orient alongside the fibre molecules 44, causing a large proportion of the dye to be present as a monolayer, which fades rapidly.

Other means by which high light fastness may be attained are the use of fibres of very high or of very low regain, and aftertreatments which make the dye completely insoluble in water.

For high light fastness in pale colours pigments should be used.

#### 8. SUBSTANCES CAUSING REDUCTION IN FASTNESS

A marked reduction in light fastness is likely to follow the use of at least three general classes of compound with certain classes of dyes—

- Hydrogen-bonding agents, e.g. phenols or urea, used during dyeing or as aftertreatments
- (ii) Cationic surface-active agents used similarly
- (iii) Any solid foreign matter introduced into the fibre before dyeing, e.g. delustrants or resins\*.

Except that sometimes a chemical action may be set up which improves fastness (cf. §§ II,2(b) and III,4 above).

It seems possible that because of (iii) a coloration produced by overdyeing a spun-coloured manmade fibre may be less fast than the same dyeing applied to an uncoloured fibre, but no information is available on this point. Perhaps also some of the so-called catalytic fading effects noted with water-soluble dyes, in which the presence of one dye tends to accelerate the fading of another on the same fibre, may be traceable to such a physical effect. That is, the internal structure of the fibre may be partly blocked by the particles of one dye, so that those of the other cannot so readily grow.

#### 9. SIGNIFICANCE OF HIGHEST GRADE

It appears that the standard grade 8 of light fastness is a natural limit which normal dyed fibres are rather unlikely to exceed. This is not merely because no higher standard is available, so that any coloration faster than 8 is bound to be classed as 8; if this were so, a high proportion of values of 8 would be found, whereas amongst the total number of assessments scrutinised for this survey-nearly 24,000-less than 30 individual values of grade 8 were found. These are all included in the azoic, metal-complex, and vat ranges, in which about 500 individual assessments were examined. The limit would appear to be set by some physical restriction to the growth of particles, imposed by the nature of the fibres themselvespresumably, in the fibres of regain above 4%, the size to which their internal pores can stretch to accommodate dye crystals. There is no such restriction to the size of particles of pigments applied as paints, etc., and consequently a much higher proportion of commercial pigmentations have a fastness of 8- in some commercial ranges the proportion is as high as 20% (Stead, D. M., private communication).

#### 10. SUGGESTIONS OF POSSIBLE TECHNICAL VALUE

The following procedures may help to produce dyeings of high light fastness—

- (a) The use of dyes which have-
  - (i) The chromophoric centre protected by chelation
  - (ii) Large planar molecules without strongly ionic groups around the periphery
  - (iii) Low solubility in water.
- (b) The use of fibres which-
  - (i) Have high or very low regain, viz. > 10% or < 1% .
  - (ii) Contain no solid adjuvant other than dye.
- (c) The use of pigments, which especially in pale colours will give better fastness than dyes.
- (d) The avoidance of hydrogen-bonding agents or cationic surface-active agents in dyeing or aftertreating processes.
- (e) The incorporation of a little oxidising agent in protein fibres or a reducing agent (e.g. a trace of formaldehyde) in non-protein fibres.
- (f) The removal of smaller particles of dye. Fading is always judged by the rate at which dye is destroyed in the earlier stages, i.e. < 50%

loss of dye; in fact, before the recent recommendations were issued <sup>54</sup> it was often judged at the first significant fade <sup>45</sup>. This means that the fastness is largely determined by the proportion of the smallest particles of dye that are present, because they fade first. Therefore to achieve high fastness it is more important to reduce the proportion of small particles to a minimum than to attempt to develop more of the largest ones.

#### 11. POSSIBLE VALUE OF CFG CURVES IN COMMERCIAL PRACTICE

In practice, if the fastness at three depths, or even at two only, were given, that at any intermediate depth could be read off by use of a simple table printed in the pattern card. Two depths at a ratio of 1:50 should have a difference in grading between 1-2 and 4 units, usually about 2-3 units, which should be enough to ensure the required accuracy in estimation. Fastness values for depths greater or less than those quoted could be similarly estimated, because it appears that the CFG curves can be considered linear for all practical purposes not only over the range used here, but also for some distance at either end.

This procedure might be found useful in selecting dyes suitable for showing "balanced" fade (i.e. each dye fading at the same rate, to ensure no change in hue). The fastness of each dye in the mixture could be estimated at the respective depths to be used, and the relative merits of alternative mixtures thus more correctly evaluated.

#### VI—Experimental; Discussion of Standards and Testing Methods

#### 1. THE LIGHT-FASTNESS STANDARDS

The fastness gradings used were determined by use of the B.S. 1006 standards  $^{52-54}$  in this country, and by those of the Echtheitskommission des Vereins Deutscher Chemiker (DEK) on the European continent. The British series differs only slightly from the Continental, several of the members of each being in fact identical  $^{53}$ .

Recently a proposed international test has been published in which the method of use of the standards has been elaborated and more precisely defined <sup>53, 54, cf. 36(b)</sup>, but it is believed that nearly all the data used here were obtained by the earlier procedure.

The test samples were exposed alongside the standards, and a fastness grade was given to each according to the corresponding standard which faded at the same rate, when judged during the early stages of fading. Samples falling between two standards were given an intermediate rating, e.g. I-2. A full account of the method of assessment used by one manufacturer has been published <sup>38</sup>, though it is probable that the actual details may differ between one manufacturer and another.

The eight standard patterns are in geometrical order of increasing fastness from 1 to 6, each differing from that next below by a factor of 2; but No. 7 and 8 are less well spaced, the ratios being greater than 2, though by how much is not known. Careful quantitative work has shown that the interval factor for No. 1-6 is 2-0 for fading to six "just perceptible steps", but that it varies

slightly for different degrees of fade. In the present work, with a few exceptions (indicated), only dyes in which all the depths have fastness numbers between 1–2 and 6 inclusive have been considered. Grade 1 figures have not been used because they may include dyes of any degree of fastness lower than 1. Table VI shows the total numbers of dyes in each class from which the choice was made.

#### 2. Possible sources of error in assessment

It is clear that errors in placing any dyed pattern on the light-fastness scale may arise from several causes, which are listed and discussed below. They may be divided broadly into two classes— (a) those connected with the conditions under which the patterns are exposed, and (b) other, miscellaneous causes.

#### (a) Variations in Conditions of Exposure\*

This is the factor most likely to affect the comparability of fading measurements made at different times or places, because both the quality of illumination and the state of the atmosphere are important in determining rates of fading. Humidity, for example, influences fading rate considerably. Hedges <sup>34</sup> carried out careful quantitative tests by which he showed that the amount of dye faded in a given time is in many cases proportional to the moisture content of the dyed fabric, and thus is related to the temperature and the relative humidity of the atmosphere (cf. also McLaren <sup>42</sup>). The B.S. 1006 standards themselves were selected from dyes least likely to be affected by changes in exposure conditions.

The possible effect of variable exposure conditions upon the data used here are discussed later.

#### (b) Other Sources of Error

(i) SEQUENCE ERRORS IN THE STANDARDS— The work of Ricketts <sup>48</sup>, just referred to, shows that errors from this cause are unimportant, at least in the range of grade numbers selected.

(ii) Errors in Visual Judgment— The assessments are made by the visual judgments of trained colourists, and it is known that such judgments are reliable of 10, 60, 62. The agreement between fastness grades given to acid dyes on wool by different observers, referred to below, is further evidence of the reliability of the judgments made.

(iii) IRREGULARITIES IN RELATIVE DEPTHS— Fastness figures are now usually given for more than one depth of colour, generally three—a medium or "standard" depth, a heavy dyeing of twice the standard depth, and a pale dyeing of one-third of the standard strength. In some dye classes different relative depths are used, as indicated in Table VI.

One set of data for azoic dyes includes the actual quantities of coupling component present on the fibre for each dyeing, but for all other dyes the factors by which depths differ refer, according to

• All the data used here were obtained by daylight exposures. Fastness figures determined in a fading lamp are often different from those obtained in daylight. This has been shown by McLaren to be due partly to differences in temperature and humidity and partly to differences in the fading efficiency of various parts of the spectrum, which vary according to the normal fastness of the dys 48, 41.

the normal convention, to the relative amounts of dye initially present in the dyebath and not necessarily to those taken up by the fibre. Nevertheless, the quantities of dyes used are well below the saturation values of the fibres <sup>58</sup>, and thus by the laws which govern adsorption processes they can be considered approximately proportional to the amounts actually present on the fibre.

In §§ II and III all the comparisons were made between pairs of dyeings of "medium" depth, except in the case of disperse dyes on Terylene and nylon, where only figures for "heavy" dyeings are available. The assumption is made that the amounts of dye required to produce the same apparent depth on the two fibres compared are the same. This assumption is unlikely to introduce any significant error even if the amounts are in some cases different. Data in Table VI show that even a twofold difference in amount of dye would introduce an error of only about one-quarter of a grade in most cases, and this is much less than most of the differences observed.

(iv) Coarseness of Interval Factor in Standards—The number of integral and intermediate grades considered here is limited to ten (between 1–2 and 6, the intermediate grades being given half-unit values), so that the three figures for any one dye are too imprecise for a curve to be drawn. In Table VI, however, results for at least ten dyes in each class (with a few exceptions) are averaged. Since each grading is considered to be given correct to about 0-5 unit, the averages should be correct to at least about 0-05 unit, and are, of course, still more precise where larger numbers are taken. In some cases over 100 dyes are included in one class, so that the average grades are then given to the second decimal place.

(v) Range of Hues—Throughout this paper the selections of dyes have been made to cover all parts of the spectrum. The exposures were made to daylight. It appears justifiable to assume that the continuous spectrum of daylight will ensure that the action of the physical and chemical factors discussed will be similar for dyes of all hues. It should not be affected by differences in the absorption spectrum of the dyes or by lack of continuity in the emission spectrum of the illuminant.

#### 3. VARIATIONS BETWEEN ASSESSMENTS MADE BY DIFFERENT MANUFACTURERS ON COTTON, SILK, AND WOOL

No direct test can be made on the present data to analyse the relative importance of separate sources of error, but a useful indirect test can be made by comparing the assessments made by different manufacturers on the same dyes. This has been done on as many as possible of the dyes whose identity can be established from the Colour Index (First Edition) and which are made by both British and Continental manufacturers. The results are shown in Table IX. The selection is limited, but covers several dye classes and a representative range of hues.

Direct dyes in almost every case are given higher ratings on the Continent than in Britain, the average difference being about 1-1 grade, but

					(Re	elative o	depths	1 1 2)						
C.I. N	0.			British					C	ontinen	tal			
or Colo	ur			(ICI)				FBy				8		
200000			_	-		CT DY		COTTON						
27950			3	3	3-4		3	4	4-5	i	-	-		
29150			2	2-3	2-3		3	3	4					-
29160 24895			3	1-2	2-3		3	3	4					-
22480			1	1	1			•	4		2	45		43
22445			1	1	1		_				1	2		2
22570		1	1-2	2	2-3						2	3		4
22610			I	1	1		1	2	3		1	1		2
22130		1	1-2	2	2-3		1	1	2		_			_
22310		1	2-3	3	3-4		2	3	4					
22311		1	1-2	2	3		2	3	4					_
22345			(2	2-3	3-4)		-	-	_		2	3		4-5
23400	1		1	1	1-2		1.	1	1		1	1		1
23710			1	1	1-2			-	-		1	2		2
23850	1		1	1	1-2		1	2	3		-	-		-
24100			1	1	1		1	1	1			-		
31970			1	1	1		1	1	2					
24410			1	1	1		1	2	2		1	1		2
35660			1	1-2	2			-	_		1	2		3
35680		1	1-2	2	2				-		1	2		3
30315			I	1	1-2				-		2	2		3
40015			3	3-4	6				-		2	3		4
19555 - Yellov			4	5	5-6						5-6 5	6		6
Scarle			ī	1-2	1-2		2	2	3			5		5-6
Red	36		4	5	5-6			-			65	6		6-7
2000				· ·								0		0-1
		C.I. No	),		Bi	itish (I	CI)		C	ontinen	tal			
					Basic	Dygs	ON SI	EK		Ciba				
		41000			1	1	2		1	1	1-2			
		42510			1	1	2		1	1	1-2			
		45170			2	2	2		1-2	2	2-3			
					Draw	T DYE	M ON S	line		Ciba				
		24895			4-5	5	6	71.8.785.	5	6	6			
		22311			2	3	3		3	3	3-4			
		30295			2	3	4		2	2-3	3			
						DYES 0								
		59825			6-7	7	7	ON	6-7	BASE 7	7-8			
		69800			7	7-8	8		7-8	7-8	8			
		69835			7	7-8	8		7	7-8				
		69815			7	7-8	7-1	4	7	7-8				
	British				(	Contine	ntal							
	(ICI)			FBy	,	/ / / / / / / / / / / / / / / / / / /	Ciba			Gy			8	
				-	Acid	DYES		OOL		-				
4	5	4-5	$\overline{}$			4	4-5		4	5	5-6	-		-
4-5	5	5-6	-	N		4	4-5	5-6	4	4-5	5-6	4	4-5	5-6
3	4	4-5	-	-	400,000	3	4	4-5	3	4	4-5	3	4	4-5
2	2-3	3	-	-	-	- Amount	-	-	2	2	3	-	-	
2	2-3	3		0.00-0	~ ~	2	2	3	-	-		2	2	3
3	4.	4	-	ACCOUNT.		3	4	4	-3	4	4	3	4	4
3-4	3	4	2	3	4	2	3	4		-		2	3	4
3	4	4-5	-	-	-	3	4	4-5			-	3	4	4.5
2	2-3	3	decree a	-		2	2	3	2	2	3	3	4	4-5
1-2	1-2	2	-			1	1	2	-	-	-3	1	I	2
1	1-2	2	_			1	1	2	1	1	2	í	1	2
2	2	3				_	_	-	_	_		2	2	3
2	3	3									-	2	2	3
2	2	3	-	*****	*	2	3	3	2	3	3	2	3	3
2-3	3	4	3	3	4	-	-	-			-		-	-
I	2	3	-		-	1	2	3				1	2	3

ratings for acid dyes on wool are almost the same. Thus, of the eighteen dyes of the latter class examined, six have the same ratings at all three depths in the British and Continental tables, and eight differ in grading only at one depth, five of these by approx. ½ grade and three by 1 grade; only one (C.I. 22890) has very different ratings in the two cases. The averages for the 18 dyes are almost identical (2.9, 3.4, 3.9; slope 1.5).

McLaren has recently described an interesting investigation into some of the effects of temperature and humidity conditions upon light-fastness assessment <sup>43</sup>, and has shown that, contrary to what at first might be expected, fading of dyes sensitive to humidity may actually be more rapid on dull than on bright days. The reason is that in bright sunshine the humidity of the air in contact with the pattern falls, because the fabric becomes hotter

TABLE X.

Summary of Differences between British and Continental Ratings for Direct Dyes

		British	Contine	ental
			FBy	8
	DYES	WITH ALL GRADINGS	LYING BETWEEN 1-2 AND 6	(TABLE IX)
(5 dyes)		2·4 2·9 3·3 (Slope 1·8)	2·8 3·4 4·1 (Slope 2·0)	
(5 dyes)	***	2·9 3·6 4·3 (Slope 1·8)		3·3 4·0 4·8 (Slope 1·9)
		DATA FOR V	VHOLE CLASS (TABLE VI)	
		2·1 2·6 2·9 (Slope 1·1)	2·13 2·65 3·05 (Slope 1·2)	2·65 3·32 3·93 (Slope 1·6)

The differences in the direct-dye ratings are conveniently considered in the form of the average data and slope values given in Table X. The selections are clearly not fully representative of the whole, but even so the same trend is obvious in these small samples as in the larger ones from which they are drawn; the Continental fastness figures are higher and their. CFG curves are steeper.

It is therefore concluded that data for dyes on wool are truly comparable even between manufacturers, but for direct dyes on cellulose comparisons should preferably be made only among each manufacturer's products.

There are insufficient data in Table IX to enable the agreement of the different ratings on silk to be correctly judged, but it appears to be intermediate between that for wool and that for cotton. The few vat dyes on cotton show little variation between manufacturers.

#### 4. CAUSES OF VARIATIONS BETWEEN ASSESSMENTS BY DIFFERENT MANUFACTURERS

The differences in the ratings for direct dyes show that the tests must have been made rather differently in the respective countries. All things considered, the variation must lie in the conditions of exposure rather than in the other factors (b, above). Indeed, as anticipated, the other factors seem to have no important influence on fading assessments, because it is hardly credible that the wool dyes could otherwise show such good agreement. They could do so only if variations introduced by one set of factors were just cancelled in each case by those arising from the other.

Dyes tend to fade more readily in atmospheres of high than in those of low relative humidity, but it is known that change in humidity affects the fading of dyes much less on wool than on cotton <sup>52</sup> (for this reason wool was chosen for the standard patterns), and it seems likely that the higher Continental ratings for dyes on cotton may be due to the tests being made under drier conditions.

than it does under dull conditions. McLaren finds, in fact, an inverse relationship between hours of sunshine during exposure and what he terms the "effective humidity", i.e. the degree of constant humidity in a closed vessel which is required to produce the same rate of fading as occurs during free exposure in the atmosphere.

TABLE XI
Relative Humidity and Temperature Data at Various
Fading Stations

Location		Period covered	Sunshine (hr.)	Mean R.H. (%)
Manchester-				
Ringway A	irport	1946 - 1955	-	81.5
Whitworth	Park	1953-1955	arrives.	80
		1921 - 1950	1071	-
Barcelona		1925-1955	-	71
Basle *		1929-1948	1659	-
		Unspecified	_	80
		Unspecified		77
Leverkusen		1903-1930	0.00	80
		1954-1955		77

Data of average atmospheric conditions for the areas of the respective fading stations at which, it is believed, the dyes under consideration were tested (Table XI) show little difference in mean relative humidity between Manchester, Basle, and Leverkusen, but there are considerably higher sunshine averages at Basle than at Manchester (no comparable sunshine data are available for Leverkusen). The average daily sunshine hours at Manchester and Basle over the whole year are 2-93 and 4-55, corresponding, in McLaren's relationship (his Fig. 2 42), to "effective humidities" of ca. 29 and 16% respectively.

A study of Hedges' results <sup>34</sup> for the fading of four basic dyes on cotton at different humidities shows that the fading varies from about one-third faster to twice as fast at 30% R.H. compared with 15% R.H., so that a fading rate of a cotton dye in



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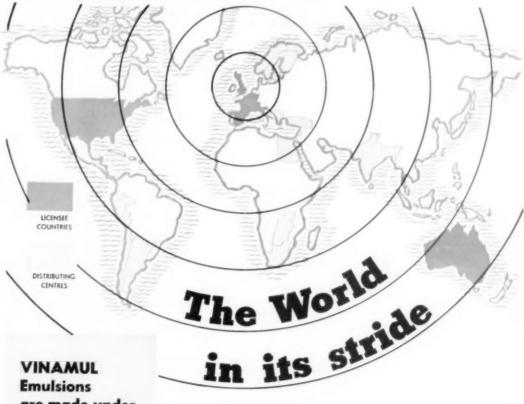


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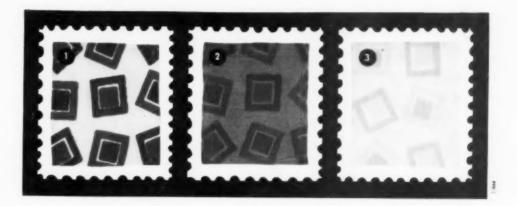
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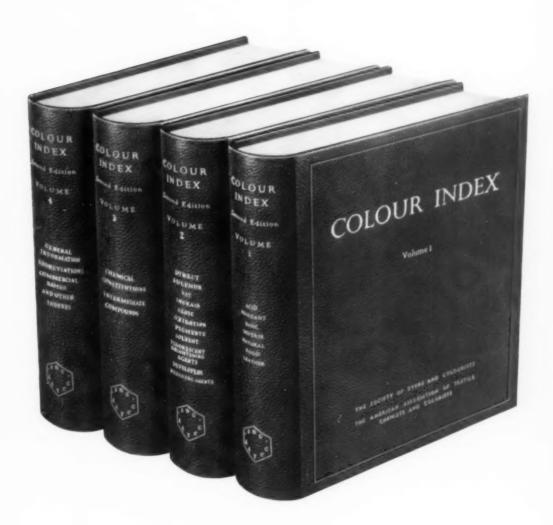
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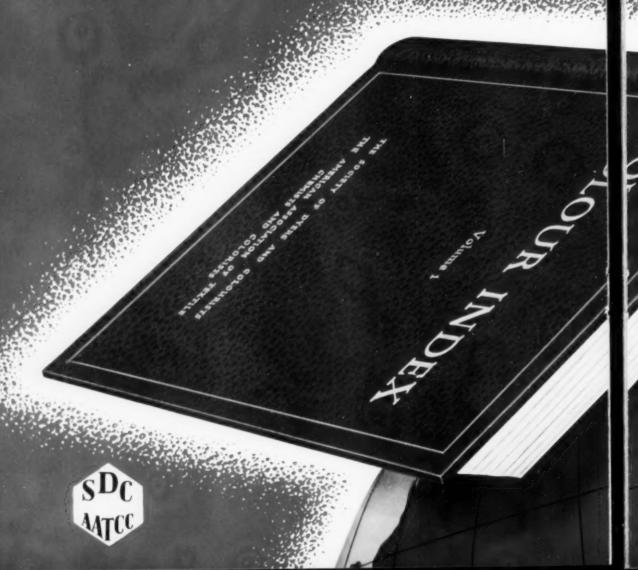


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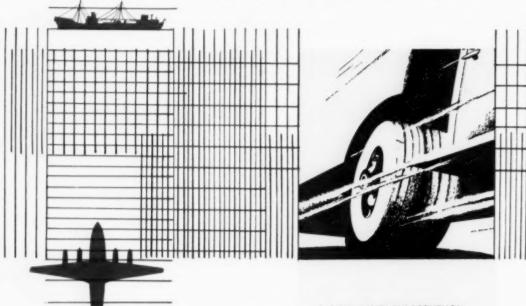
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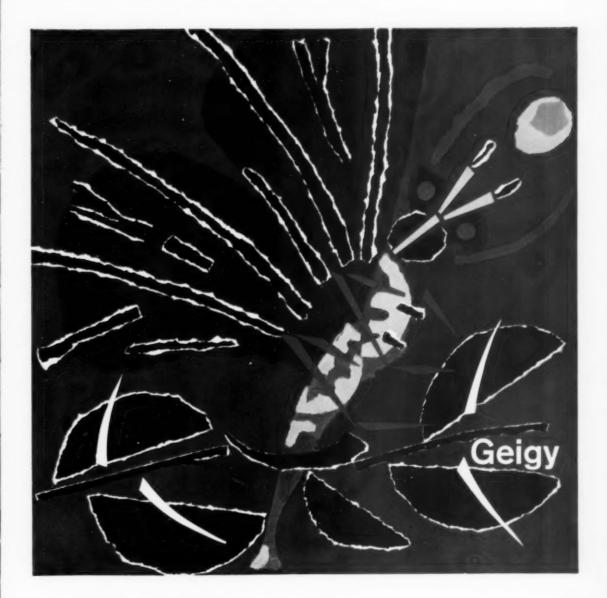
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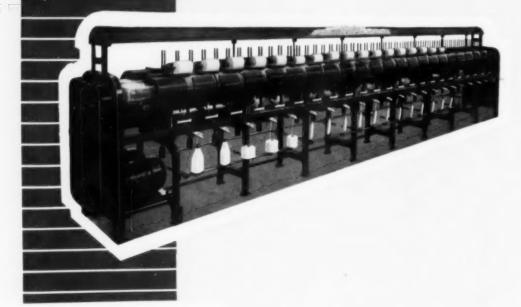


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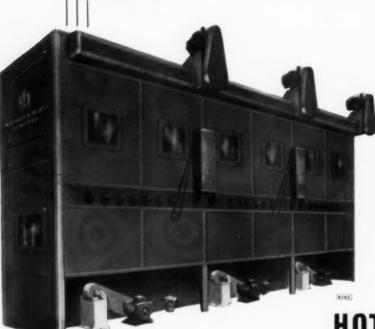
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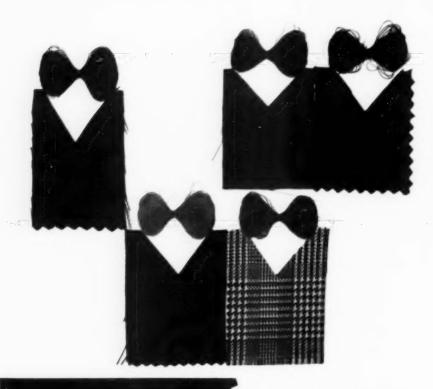
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Basle one-half that in Manchester (i.e. one grade higher) can quite reasonably be attributed to the difference in hours of sunshine.

### 5. VARIATIONS BETWEEN ASSESSMENTS ON CELLULOSE ACETATES

As already discussed (p. 148), it may be expected that fading on certain man-made fibres, e.g. cellulose triacetate, will be rather sensitive to humidity, and hence to the amount of bright sunshine during exposure. There is not much evidence on which to check this point, but two possibly significant facts emerge from the present data—(a) The relative differences between dyes on pairs of fibres tends to vary more from one manufacturer to another when one at least of the fibres has low than when both have medium or high regain. (b) Fastness data obtained by two firms on an identical series of dyes on secondary cellulose acetate and cellulose triacetate are different (Table XII).

do, give curves differing from the average. Therefore, as the CF curve is illustrative of certain properties of a single dye–substrate system, so it may be inferred that the present curves illustrate analogous properties associated with the class as a whole. Secondly, the ordinates differ: the CF ordinates are log  $t_{\rm F}$ , where  $t_{\rm F}$  is the time required for a given percentage loss of initial dye concentration, whereas the CFG ordinates are the light-fastness grade numbers.

To demonstrate an identity between the two families of curves it is therefore necessary to show that the light-fastness gradings of a series of depths of a dye are equivalent to  $\log t_F$  values, i.e. that they represent the times required for the loss of a given proportion of original dye, irrespective of the initial amount present.

It seems likely on first principles that the Weber-Fechner law will apply to estimates of fading. There is, in fact, some earlier experimental evidence of its applicability to visual assessments

 ${\rm T_{ABLE}~XII}$  Light Fastness of Identical Series of Ten Disperse Dyes on (Bright) Acetylated Cellulose Fibres

Mean Fastness					Tested	Year	Mean	Effective
Cell	Secondary Cellulose Cellulose Triacetate Acetate (Courpleta)		by at			Daily Sun-hr. (JanSept. inclusive)*	Humidity ** (%)	
5.6+ (Ser.	6·I+ aceta)	3.7	4.6	Courtaulds	Droylsden (Manchester)	1954	2.78	27
		4.2	5.4	ICI	(?) Blackley (Manchester)	$1955 \uparrow$	4.12	18
4.5	5-1			ICI	Detail	s not known		

<sup>.</sup> Whitworth Park, Manchester,

The Courtaulds exposures were made during the wet, dull summer of 1954; it is assumed that the ICI data were obtained during 1955, which was sunny. If this is so, then an increase in sunshine, and consequent decrease in "effective humidity" have, in accordance with McLaren's findings. decreased the fading rate on cellulose triacetate, more particularly at the greater of the two depths. Nothing definite can be said regarding the lack of agreement between the figures for secondary acetate, because the date and the atmospheric conditions of the ICI tests are not known. The differences might be attributable partly to differences in the depths of dyeing employed by the two testing stations, and partly to different physical characteristics in their supplies of fibre (both used the same triacetate), or even to differences in dyeing methods.

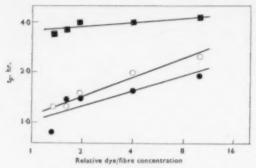
### 6. RELATIONSHIP OF CFG CURVES TO CF CURVES

The virtual linearity of the CFG curves is established by statistical analysis (Appendix 2, below). It is then desirable to consider their exact relationship to the CF curves described by Baxter et al.\*7. The two types of curve differ in two respects. Firstly, the CF curves are determined for single dyes, whereas the CFG curves represent the average characteristics of whole dye classes, individual members of which may, and indeed often

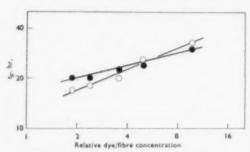
of dyed fabrics, in that Vickerstaff and Walls and describe results which they consider probably show the operation of the law in judgments by trained colourists of hue, strength, and brightness differences. Even if a dye becomes duller as well as weaker on fading, as many dyes do, the law should still hold, for Vickerstaff, White, and Waters 62 found that highly skilled colourists judge the strength of dyed patterns with equal consistency whether dullness is present or not. One manufacturer's procedure in assessing fading was stated to be "to ignore shade change and assess the pattern on rate of fading so far as loss of depth alone is concerned" <sup>28a</sup>. If the law is applicable to normal fading assessments, it follows that the percentage gain of reflectance which has occurred when a colourist judges that a significant fade has appeared in a pattern is the same for all initial values of dye content. There is a linear relationship between the logarithm of reflectance and the logarithm of dye content 7, 21, and the significant fade would thus represent the loss of a constant proportion of original dye content at any depth of

This conclusion has now been checked experimentally on three dye-fibre combinations (Fig. 20–23) chosen more or less at random, and the results are in reasonable agreement with the hypothesis, because the CF curve (for 5% fading)

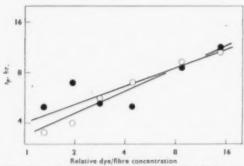
<sup>†</sup> Cf. date of publication 2786



Fto. 20—Chlorazol Sky Blue FF on Viscose Rayon



Fto, 21— Chloragol Fast Helio 2RK on Viscose Rayon



Spectrophotometric measurements, 10% loss of initial dye concn.

Spectrophotometric measurements, 5% loss of initial dye concn.

Visual estimate of just perceptible fade

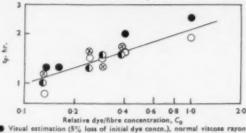
tp - Time required for chosen degree of fade

Fig. 22-Acilan Fast Green 10G on Silk

Fig. 20–22 — Comparisons of Visual and Spectrophotometric Fading Measurements

measured spectrophotometrically is in each case close to that plotted from visual estimates of fading on the same patterns. Since this effect is the result of the operation of purely optical effects, and is unlikely to be influenced by the chemical nature of the dye or the substrate, it is assumed that it is applicable to all the dye-fibre systems examined here.

The CFG curves are, therefore, probably formally identical with CF curves, except that they are determined, not by the specific properties of a single dye, but by the average properties of a whole class of dyes applied to a given fibre.



Visual estimation (5% loss of initial dys contn.), normal viscose rayon
Visual estimation (5% loss of initial dys contn.), "reduced-imbibition"

Spectrophotometric measurements, normal viscose rayon
 Spectrophotometric measurements, "reduced-imbibition" rayon

Fig. 23 — Comparison of Visual and Spectrophotometric Fading Measurements with Chloragol Sky Blue FF (ICI) on Viscose Rayon

The experimental details of the above test are given below.

### Comparison of Visual and Instrumental Determination of Fading Rates

The data in Fig. 20-23 were obtained as already described 7, 15. Adsorbed dye was determined by extraction with pyridine, and reflectance values were obtained spectrophotometrically by means of the Unicam SP 500 instrument. Exposures were made with a 400-watt Osira (G.E.C.) mercuryvapour lamp. Visual judgment of fading was made (by two observers) at frequent intervals until a quite distinct loss of dye had occurred, the time  $t_F$  being recorded at which a just perceptible loss was noted. The  $t_F$  values for reflectance measurements refer to the appropriate reflectance increases corresponding to an actual loss of 5% or 10% dye, as indicated, the relationship between reflectance and dye content being determined experimentally. The curves for the 5% loss are less reproducible than those for 10% owing to the difficulty of making precise measurements of fading rates in the very early stages.

The viscose rayon fabrics were identical with those used before  $^7$  (Series I). The silk fabric had the following specification—Weave, plain; warp and weft, 80 denier, 35–40 filaments; set  $112 \times 112$ ; 1.5 oz./sq.yd.

The dyes used were of normal commercial quality and were applied by the usual methods.

### VII- Appendixes

### 1. PREVIOUS WORK ON THE RELATIONSHIP BETWEEN DYE CONCENTRATION AND FADING

It is well known that the light fastness of a dye on a textile fibre generally increases with increase in depth. The first investigators to study this phenomenon in any detail were probably Barker, Hirst, and Lambert in 1927. Using wool fabrics dyed with acid dyes and measuring fading by means of the Lovibond Tintometer, they found that the amount of dye faded after a given exposure to light is approximately constant whatever the initial concentration, i.e. that the proportion faded falls regularly with increase in concentration. This, of course, is a characteristic of zero-order fading, which implies the fading of large aggregates. If the dye were present principally in molecularly

disperse form and all its molecules were equally accessible to oxygen and moisture, a first-order reaction would be expected. The same authors also observed an empirical relationship between the amount of dye faded and the square root of the time of exposure. Cunliffe and Lambert <sup>22</sup> (1932), also using wool dyed with acid dyes and employing similar measuring techniques, examined these relationships in some detail and established a more complex one between the extent of fade after a given time and the initial dye concentration. These researches formed part of the work leading to the establishment of the British light-fastness standards (B.S. 1006).

Cunliffe and Lambert found that an empirical relationship—

$$F = m\sqrt{t + k} \tag{i}$$

(where F is the quantity faded after time t, and m and k are constants) was applicable to most of the dyeings they examined, from values of fading of a few per cent up to about 60%; but the relationship—

$$F = a \log t + b \tag{ii}$$

(a and b being constants) was followed rather more satisfactorily than (i) for values of fading from about 25% upwards. They considered that the relationship (i) is somewhat accidental, the fundamental equation being—

$$F = 100 (1 - e^{-kt})$$
 (iii)

Cunliffe and Lambert also observed, again empirically, that the relationship between quantity of dye faded and initial dye concentration  $C_0$  is represented by—

$$F = -d \log C_0 + f \tag{iv}$$

(d and f being constants). Finally they combined the separate equations into one relating fading after time t ( $F_t$ ) to time and initial concentration—

$$F_I = 100 \left[1 - e^{-t(a \log C_0 + b)}\right]$$
 (v)

It is clear from a consideration of equations (ii) and (iv) that there should be a linear relationship between  $\log C_0$  and  $\log t$  for a given amount of fading. This is in fact the one established spectrophotometrically by Baxter  $et~al.^7$  for the fading of fabrics and transparent films and shown here to hold for visual estimations also. The only difference between Cunliffe and Lambert's relationship and that observed here is that these workers found that equation (ii) represented amounts of fading above about 25%, whereas the relationship established here has been observed for fading at 10% loss of dye and less.

### 2. STATISTICAL TREATMENT (By John C. Eaton)

In any particular class of dyes there are considerable differences of light fastness from dye to dye. Only the *slope* of the regression line, however, is required in the present investigation; hence for each dye deviations from the mean light fastness of the three given values were used in calculating the regression coefficient. This eliminated one variable factor, the intercept. The slopes obtained by this method differ very little from those found

directly from the original figures, but the variance of the regression is very much reduced, permitting a more sensitive test for difference of slope between one group of dyes and another. Substantially the same slopes are obtained by the easier method, adopted in Fig. 14, 15, and 19, of fitting the regression line visually to the mean grades at each concentration level.

In all groups of dyes the relative concentrations are constant throughout, and it is thus permissible to average their logarithms. Alternatively, the data can be referred to an arbitrary scale, the lowest concentration being taken as unity, viz. 1, 3, and 6 in nearly all cases. All dyes in any group then have 1, 3, and 6 concentration units on this scale.

It is important to determine whether the data for each dye class can be adequately represented by a linear regression; or, if a curvilinear regression is necessary, whether the gradient of the curve varies much over the range considered. It is clear from the graphs that the points lie very nearly on straight lines, but it is possible that, on account of the large number of observations, a curvilinear regression might still be theoretically required, even if it departed very little from linearity over the given range. On testing the results for Setacyl Direct dyes on cellulose acetate 29c (a class having almost the smallest number of representatives of any of those selected) for linearity by the usual analysis-of-variance test, slightly modified (see below), it was found that departure from linearity was not significant even at the 10% level of probability; i.e., roughly speaking, if the population were truly linear, then samples of 45 observations drawn from it would deviate from linearity to the extent of this sample, or more, as frequently as once in 10 times.

As a further check, a parabola was fitted exactly to the means of the light-fastness values for the three given values of the logarithms of the relative concentrations of these dyes. The greatest and least gradients from the parabola in the experimental range gave slopes of 2·1 and 1·6 respectively, as compared with the value of 1·9 obtained from the regression line.

In order to test whether the slopes of the regression lines found for the different groups of dyes really differ from each other, some pairs were selected for investigation by the t-test. Direct dyes on cotton (68 dyes) 20s were compared with vat dyes on cotton (18 dyes) 37d, because there is a large difference in slope between these two groups (1.1 and 1.8 respectively). If there were no significant difference here, it would be unlikely that any other pair would give a significant difference. The result, t = 6.883 (256 degrees of freedom), is highly significant, implying that there is a genuine difference between these slopes. The same series of vat dyes on cotton 37d was then compared with Setacyl Direct dyes on cellulose acetate (15 dyes) 29e, because here, not only are the slopes (1.8 and 1.9) fairly close, but also the numbers of observations  $(3 \times 18 \text{ and } 3 \times 15)$  are amongst the smallest of the groups examined. Both these factors make the test as exacting as possible; i.e. if these differ

significantly, all the others ought to differ significantly also. In fact t is found to be  $1 \cdot 280$ , which is barely significant, for P is slightly greater than  $0 \cdot 1$ .

Thus, the fact that even the most unfavourable pair shows a difference which almost attains significance at the 10% level makes it virtually certain that there is a definite relation between class of dye and slope.

In order to make this description more definite, the calculations relating to Setacyl Direct dyes on cellulose acetate are given in greater detail. The actual data given in the original publication  $^{29c}$  are quoted in Table XIII. The mean for the first dye, viz.  $(3+4+4\cdot5)/3$ , is 3·83, which gives deviations from the mean of the three values of -0.83, +0.17, and +0.67 respectively. To eliminate the negative numbers, and to simplify plotting of the points when required, the constant 4·0 was added to each deviation, giving the data in the last three columns of Table XIII.

TABLE XIII

### Selected Setacyl Direct Dyes on Cellulose Acetate

Detretted Settle).			3				
Setacyl		astne		Modified Fastness			
Direct		rades		Grades			
Dye Dspth	3	1	2	La	3.	6 e	
Yellow B8	3.0	4:0	4-5	3.17	4-17	4-67	
Orange 4R	4.0	5-0	6.0	3.0	4.0	5-0	
Scarlet B	4.5	6.0	6.0	3.0	4.5	4.5	
Red BN	4:0	5.0	6-0	3.0	4.0	5.0	
Pink 3B	5.0	6-0	6-0	3.33	4.33	4.33	
Violet 4RT Supra	3.0	4.0	4.5	3-17	4-17	4-67	
Violet R	4.0	5.0	5-5	3.17	4-17	4.67	
Violet B	3.0	3.5	4-5	3-33	3.83	4.83	
Blue BM	4.5	5.0	5.0	3.67	4-17	4-17	
Blue 2G8	2.0	3.0	4.0	3.0	4.0	5-0	
Blue Green BS							
Supra	4.5	5.0	5.0	3-67	4.17	4-17	
Navy Blue GBS	4.0	4.5	5.0	3.5	4.0	4.5	
Navy Blue CGN	4.0	4.5	5.0	3.5	4.0	4.5	
Black BMS	3.0	4:5	5.0	2.83	4.33	4.83	
Black GM	4.0	5.0	5-5	3.17	4-17	4-67	

· Arbitrary scale.

Taking X as the logarithm to base 10 of the concentration on the arbitrary scale, and Y as the modified fastness grade, while in the usual way—

$$x = X - X$$
 and  $y = Y - \overline{Y}$ 

 $(\overline{X} \text{ and } \overline{Y} \text{ are the means of the } Xs \text{ and } Ys \text{ respectively), we obtain—}$ 

 $\Sigma y^4 = 17.5003$   $\Sigma x^4 = 4.6194$   $\Sigma xy = 8.3471$  and hence the coefficient of regression—

$$b = \frac{\Sigma xy}{\Sigma x^2} = 1.807$$

which is the slope of the best straight line. The variance of b—

$$V(b) = \frac{\Sigma y^2 - b\Sigma xy}{(N-2)\Sigma x^2} = 0.01217 \text{ (43 degrees of freedom)}$$

where N is the number of sets of results. In the same way b' for direct dyes on cotton is found to be 1-078, with variance—

$$V(b') = 0.00378$$
 (202 degrees of freedom)

For the purposes of statistical analysis, however, we have assumed that the fastness n depends on two factors, one (g) characteristic of a particular dye, but independent of concentration, and one

(say  $h=a\log C_0$ ) which varies with initial concentration  $C_0$  but does not vary from dye to dye within one class. Thus for two dyes A and B in one class, at three levels of concentration, we may write—

$$n_{A_1} = g_A + h_1 + R_1$$
  
 $n_{A_2} = g_A + h_2 + R_2$   
 $n_{A_3} = g_A + h_2 + R_2$  (vi)

$$n_{B_1} = g_B + h_1 + R_4$$
  
 $n_{B_2} = g_B + h_2 + R_5$   
 $n_{B_3} = g_B + h_3 + R_6$  (vii)

where  $R_1$ ,  $R_2$ ,  $R_3$ , etc. are residuals. Since  $g_{\Lambda}$  and  $g_{\rm B}$  do not affect the slope of the line, we remove them by subtracting—

$$n = \frac{n_1' + n_2 + n_3}{3}$$

from  $n_{A1}$ ,  $n_{A2}$ ,  $n_{A3}$  in turn, giving—

$$n_{A_1} - \overline{n}_A = h_1 - \overline{h} + \frac{2R_1}{3} - \frac{R_2}{3} - \frac{R_2}{3}$$

$$n_{A_2} - \overline{n}_A = h_2 - \overline{h} + \frac{2R_2}{3} - \frac{R_2}{3} - \frac{R_1}{3}$$

$$n_{A_3} - \overline{n}_A = h_2 - \overline{h} + \frac{2R_2}{3} - \frac{R_1}{3} - \frac{R_2}{3}$$

and similarly for  $n_{B1} - \overline{n}_B$ , etc. (we have referred to  $(n - \overline{n})$  as X). The variance V(b) we have obtained is thus the variance, not of R, but of the linear function—

where 
$$a=\frac{2}{3}$$
 and  $\beta=\gamma=-\frac{1}{3}$ . Hence—
$$V(b)=[(\frac{1}{3})^3+(\frac{1}{3})^2+(\frac{1}{3})^3]\times V(R)=\frac{2V(R)}{3}$$

To test whether the slopes b, b' come from the same parent population we find—

$$t = \frac{b' - b}{\sqrt{[V(R') + V(R)]}}$$

$$= \frac{b' - b}{\sqrt{\left[\frac{3V(b')}{2} + \frac{3V(b)}{2}\right]}}$$

$$= \frac{0.816 (b' - b)}{\sqrt{[V(b') + V(b)]}}$$

Here t = 4.710 with (43 + 202 = 245) degrees of freedom. This gives the value of P as much less than 0.001. Thus there is evidence here of a real difference between the slopes associated with Setacyl Direct dyes and this series of direct cotton dyes. Other pairs are treated in the same way. While this analysis confirms that the mean slopes differ significantly from group to group, it does not necessarily follow that the assumption of a constant value of h from dye to dye within each group is thereby proved. Variations in h within a group might indeed be expected, but the analysis shows that they are of minor importance compared with the average variations from group to group. If h does in fact vary within the group, this variation goes to increase the residuals (R, etc. above), which, since it makes the test less sensitive, adds further weight to the conclusion here demonstrated. for the differences between groups are significant in spite of it.

### 3. FASTNESS DATA ON SECONDARY CELLULOSE ACETATE AND CELLULOSE TRIACETATE

Two series of data were supplied by Courtaulds Ltd. The series are directly comparable. The yarns were bright Seraceta and bright Courpleta of similar denier, dyed to similar depth and exposed side by side to daylight at Droylsden during the summer of 1954. The Seraceta was dyed at 75°c. and the Courpleta at 95°c., both from soap solution (5 g./litre). The figures are set out in Table XIV.

TABLE XIV

Light-fastness	Data	on	Seraceta	and	Courpleta
	(Bot	h fib	res bright)		

	(Bo	th fi	bres	bright)		
Dye			Cellulose Triacetate		Secondary Cellulose Acetate	
			Pale	Medium	Pale A	
Cibacet (Ciba)-	-					
Blue 2R			4-5	4-5	6	$6 \pm$
Blue B			3-4	4-5	6	6+
Sapphire Blue		***	3	4	3-4	4-5
Turquoise Blo	ue G	***	3-4	-	5-6	-
Scarlet BS	***	***	2 - 3	-	4	4
Violet 5R	***	111	5	-	6	-
Yellow GN	***	***	4-5	5	5	6
Duranol (ICI)-						
Brilliant Blue	BN		2	3	4	5
Blue G	***	***	3	3-4	5	5 - 6
Blue 2G	***		3-4	4-5	5-6	6
Red 2B		***	4	5-6	6	6-7
Red X3B			3-4	4-5	5-6	6
Violet 2R	***	***	.5	5-6	6	6 - 7
Dispersol Fast (	ICD-					
Yellow G			4-5	5	6	6-7
Orange G			4	5-6	5-6	6+
Scarlet B			3-4	4	5-6	6
Orange B			4	4-5	6+	6+
Crimson B	***	***	3	4	5-6	6
Celliton Fast	Blue F	FG				
(BASF)	+1+	***	4-5	5	6	6 +
Serisol (YDC)-						
Dark Blue B			2-3	3-4	3	3-4
Fast Yellow			4-5	6	6+	6+
Fast Red 3B	L		5	-	6	-
Eastman (TE)-	_					
Fast Yellow			6+	$6 \pm$	6+*	6+
Fast Yellow		***	6+	6 +	6+0	6+
Fast Brillian	t Red					
2BGLF			4	-	5-6	-
Blue BNN	***	+**	3-4		5	-
Fast Red GL	F		5	6	6+	6+
Fast Violet 3	RGLF	***	5	5-6	6	6+
Interchem Acet	ate (IC	)_				
Blue RLF 40			3-4	4	4	5
Yellow RLF		***	6	6+	6+	6+
Celanthrene (De	(P)					
Violet BGF			5-6	5-6	6+	6+
Fast Yellow		***	6+		6+*	6+
rase renow		***	14			4

<sup>·</sup> Fastness marginally better on Seraceta, but not assessed.

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Farbentabriken Dayer

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- (dd)
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### Tentative Specification for the Larval Testing of Mothproofed Wool Serge\*

INTERNATIONAL WOOL TEXTILE ORGANISATION

### Introduction

In January 1951 the Technical Committee of the International Wool Textile Organisation set up a subcommittee to devise a reasonably accurate method for the larval testing of mothproofed wool. The basis of the initial discussions was the Swiss Association for Testing Materials Specification SNV 95901 and a provisional method devised by British laboratories collaborating under the aegis of the Society of Dyers and Colourists and the Textile Institute†. The following members have actively collaborated in the work of the Subcommittee—

	GREAT BRITAIN					
Mr. J. Barritt	Wool Industries Research Association, LEEDS					
Dr. E. A. Parkin	Pest Infestation Laborator SLOUGH					
Mr. P. T. Gale	Imperial Chemical Industries Ltd., Manchester					
Dr. G. E. P. Box	Imperial Chemical Industries Ltd., Manchester					
Dr. O. Davies	Imperial Chemical Industries Ltd., MANCHESTER					
Mr. S. L. Anderson	Wool Industries Research Associa- tion, LEEDS					
Dr. R. Burgess	Geigy Co. Ltd., MANCHESTER					
Mr. A. Roberts	Imperial Chemical Industries Ltd., Manchester					
	SWITZERLAND					
Prof. Dr. A. Engeler	Eidgenössische Materialprüfungs- und Versuchsanstalt, St. Gallen					
Dr. O. Wălehli	Eidgenössische Materialprüfungs- und Versuchsanstalt, St. Gallen					
Dr. R. Zinkernagel	J. R. Geigy A.G., BASLE					
	GERMANY					
Prof. Dr. H. Zahn	Chemisches Institut der Universität, HEIDELBERG					
Dr. W. Frey	Biologische Bundesanstalt für Land- und Forstwirtschaft, Institut für Vorratsschutz, Berlin-Dahlem					
Dr. E. Laibach	Farbenfabriken Bayer, Leverkusen					
	NETHERLANDS					
Dr. G. J. Schuringa	Centraal Laboratorium T.N.O.,					
Dr. H. J. Hueck	Centraal Laboratorium T.N.O.,					

Delet

This specification is being published also in other periodicals.
Copies in English, French, and German are obtainable from the
International Wool Secretariat, 18-20 Regent Street, London S.W.I.

DELFT

Dr. A. D. J. Meeuse formerly

Vezelinstituut T.N.O.,

The following laboratories undertook larval testing—

Biologische Bundesanstalt für Land- und Forstwirtschaft, Institut für Vorratsschutz, Berlin-Dahlem, Germany

Centraal Laboratorium T.N.O., Delft, Holland

Eidgenössische Materialprüfungs- und Versuchsanstalt, St. Gallen, Switzerland

Farbenfabriken Bayer A.G., Leverkusen, Germany

Geigy Co. Ltd., Manchester, England

J. R. Geigy A.G., Basle, Switzerland

Imperial Chemical Industries Ltd., Manchester, England Pest Infestation Laboratory, D.S.I.R., Slough, England Wool Industries Research Association, Leeds, England

Dr. Schuringa was elected Chairman and secretarial duties were performed principally by Dr. Hueck. The Subcommittee has met eight times and this tentative specification is based on their consideration of over 50 reports, mostly on experimental work performed by the members.

Detailed tests of existing methods made it clear that variability among the results obtained by different laboratories carefully carrying out a specified technique was too great and that attention must primarily be directed to reducing this interlaboratory variation. Retaining weight loss as the basic criterion of damage, the introduction of reference standards of serge impregnated with known levels of a suitable mothproofing agent has enabled the damage to be assessed in terms of the percentage impregnation of the reference agent.

Trials based on this principle have shown a reduction of interlaboratory variation to a level considered acceptable, and further improvement has been brought about by attention to the method used for cleaning samples after exposure to insects.

Investigation of other chemicals as possible standard mothproofing substances is being undertaken in case one can be found even more suitable than "D.A.N."; the extension of the method to fabrics other than serge is also being actively pursued.

### Tentative Standard Method of Test and Assessment for Proofness of Wool Serge against the Common (Webbing) Clothes Moth Tineola bisselliella (Hum.)

FOREWORD

The following method was evolved as a result of a large number of co-operative experiments carried out by British and Continental laboratories since 1951. The material used in these experiments was wool serge but it is hoped to extend the methods to other important wool materials when further tests are completed. The mothproofness of the material under test is assessed as the equivalent amount of a standard mothproofing compound, which may then be related to an acceptable level

On the initiative of the Society a joint committee with the Textile Institute was set up in 1948 under the chairmanship of Mr. B. J. Hannay. A provisional specification was prepared, but while work was in progress the I.W.T.O. became interested in the subject, and the joint committee collaborated in the work which led to the present tentative specification.

of this compound. Suggestions for an acceptable level are given in Appendix 4.

### I. SCOPE

This method applies to the determination of the resistance to attack of Botany wool serge by larvae of the common (webbing) clothes moth, *Tineola bisselliella* (Hum.).

The results obtained relate only to the resistance to attack of the combination of serge and its mothproofing treatment.

### 2. DEFINITIONS

(i) Standard patterns are circular patterns 4 cm. in diameter cut from fabric treated with 2:4-dinitro-1-naphthol (henceforth referred to as "D.A.N.") as described in Appendix 2. The fabric for these standard patterns shall be of wool, smooth finish, weighing between 200 and 240 g./m.² made from wool fibres of average fibre diameter not greater than 24  $\mu$ . It should be undyed, scoured but not otherwise wet-treated, with a pH of a water extract in the range 7–9 when determined according to the I.W.T.O. standard method <sup>1</sup>. Such a fabric would be made from twofold warp and weft yarns of resultant count 29 g./km. with approx. 30 warp threads per centimetre and 33 weft threads per centimetre.

(ii) Regain controls—Additional test specimens and standard patterns are used as regain controls. \*\*

#### 3. PRINCIPLE

A set of test specimens is exposed to larval attack under specified conditions of exposure side by side with two sets of standard patterns containing 0.4% and 1.2% of D.A.N. The equivalent concentration of D.A.N. which would give the same weight loss as that found for the test specimens is estimated from the results.

### 4. TEST SPECIMENS

The test specimens shall consist of circular patterns 4 cm. in diameter; these patterns must be distributed throughout the whole of the available material. Ten test specimens are required for a single assessment.

### 5. APPARATUS

- 5.1 Provide the following ancillary equipment—
  - (i) A balance capable of weighing up to 10 g. with a sensitivity of at least 0·2 mg.
  - (ii) A room or cabinet capable of maintaining the conditioning and testing atmosphere as described in § 6.2
  - (iii) A stamp of diameter 4-0 ± 0-15 cm. for punching circular patterns of fabric
  - (iv) Aluminium containers to the dimensions given in Appendix 1\*.
- 5.2 Cut five standard patterns† from each of the two treated fabrics described in Appendix 2.
- 5.3 Cut one regain control from the material under test and one regain control from each of the two treated fabrics.

5.4 Provide larvae of *Tineola bisselliella* (Hum.) between 21 and 28 days old\*, the eggs having been collected over a period of two days. These larvae must give a mean weight loss on the untreated wool serge (see § 2.1) of at least 35 mg. when tested by the standard method.

### 6. CONDITIONING AND TESTING ATMOSPHERE

6.1 CONDITIONING— The test specimens, standard patterns, and regain controls shall be in equilibrium with the atmosphere of the weighing room before weighing.

6.2 Testino Atmosphere— The test specimens, standard patterns, and regain controls shall be maintained at an average temperature within the range 23–25°c. controlled to  $\pm$  0.5°c. and an average relative humidity within the range 65–70% controlled to  $\pm$  3% R.H.

### 7. TEST PROCEDURE

7.1‡ After conditioning, weigh each test specimen, standard pattern, and regain control to an accuracy of 0·2 mg. The order of weighing shall be random, except that the regain control shall be weighed immediately before and after the corresponding series of standard patterns or test specimens.

7.2 Collect in a suitable container and free from adhering particles at least twice as many larvae as will be required for the test. Select 15 larvae as uniform in size as possible from different positions in the container such that the total weight of the 15 larvae lies between 13 and 17 mg. The larvae shall be handled in such a way as to avoid the possibility of injury. Continue similarly selecting groups of 15 larvae so that 20 groups are available.

7.3 Using groups of 15 larvae as described in § 7.2 and test containers as detailed in Appendix 1, assemble the following—

Ten test containers in each of which are placed a test specimen and then one group of larvae

Five test containers in each of which are placed one standard pattern containing 0.4% D.A.N. and then one group of larvae

Five test containers in each of which are placed one standard pattern containing 1.2% D.A.N. and then one group of larvae

Three test containers in each of which is placed one regain control only.

7.4 Put the lids on the containers and place in the testing atmosphere as defined in § 6.2 for 14 days, so that each regain control is placed next to its corresponding set of test specimens or set of standard patterns, and remains so throughout the whole of the test.

7.5 At the end of the exposure period remove carefully the insects, excrement, and silk from each test specimen and standard pattern by means of sharply pointed forceps, but include loose fibres

Containers to this specification are manufactured by Gebr. Hoffman, Thun, Switzerland.

<sup>†</sup> All patterns must be handled so as to avoid contamination, e.g. with clean forceps or clean rubber gloves.

 $<sup>^{\</sup>bullet}$  A suitable method describing the rearing of the larvae is given in Appendix 3.

<sup>†</sup> Equilibrium may be deemed to have been reached when successive weighings, at intervals of 1 hr. with the material freely exposed to moving air, differ by less than 0-1%. This equilibrium is normally achieved in 24 hr.

The test procedure may be shortened by difference weighing between regain control and test specimen using a two-pan balance.

not spun in the silk in the weight of the test specimens and standard patterns. Note for record the number of larvae alive, dead, or pupated on each test specimen and standard pattern, any differences in appearance or activity from the control larvae, and any features of the visible damage. Condition the test specimens, standard patterns, and regain controls as described in § 6.1 and weigh to an accuracy of 0.2 mg.

### 8. EXPRESSION AND ASSESSMENT OF RESULTS

Calculate the corrected weight loss of each test specimen and standard pattern as-

L = (A - B) - (C - D)where A = weight of test specimen or standard pattern before exposure; B = weight of testspecimen or standard pattern after exposure; C = weight of corresponding regain control before test; and D = weightof corresponding regain control after test.

Calculate the corrected weight losses for the set of test specimens and for the two standards and determine the cube roots of these quantities from

Let  $S_1 = \text{mean of the}$ cube roots for 0.4% of D.A.N.;  $S_2$  = mean of the cube roots for 1.2% of D.A.N.; T = mean ofthe cube roots for test

specimens. Calculate mean of standards-

$$m=\frac{S_1+S_4}{2}$$

Calculate slope of standards—

$$b = \frac{S_2 - S_1}{0.8}$$

(Note that the value of b is negative.)

The equivalent concentration of D.A.N. is then calculated and reported as-

$$P = 0.8 - \frac{(m-T)}{h} \%$$

8.1 The approximate standard error (S.E.) of P is given by-

S.E. 
$$=\frac{s}{b}\left[1+1\left(\frac{m-T}{b}\right)^2\right]^{\frac{1}{b}}$$

where s is the estimated standard deviation.

The approximate limits within which the true value lies with 95% probability are then-

$$P\pm 2$$
 S.E.

The quantity s is the square root of the experimental error variance s2, which is given by-

$$s^2 = \frac{1}{17}$$
 (Corrected sum of squares for test specimens

corrected sum of squares for 0.4% D.A.N. corrected sum of squares for 1.2% D.A.N.)

where, if E denotes the cube roots of the corrected weight losses for N test specimens and E the average of these cube roots, then the corrected sum of squares for test specimens is-

$$\Sigma E^{q} - NE^{q}$$

the corrected sum of squares for standards being calculated similarly.

### Appendix 1

TEST CONTAINER

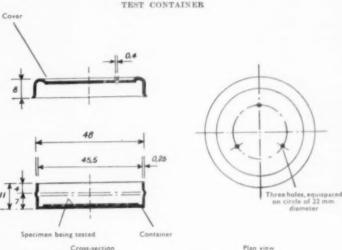


Fig. 1— Test Container (dimensions in mm.)

### Appendix 2

TREATED SERGE FOR STANDARD PATTERNS

Officially prepared serge is available from The The International Wool Textile Secretary, Organisation, Commerce House, Cheapside, Bradford I, Yorkshire. Although laboratories may prepare their own material for day-to-day use according to the instructions in Appendix 2, only use of the official standard patterns will yield results officially recognised as obtained in conformity with this Specification.

The treated serge from which the standard patterns are cut shall contain (i) 0.4%, (ii) 1.2% of D.A.N., expressed on the weight of serge at normal regain. The D.A.N. used for treating the serge shall conform to the following-

Melting point ... ...  $135-138^{\circ}$ c. Sulphated ash ... ... Not more than 0.2% Volatile matter (at  $100^{\circ}$ c.) ... Not more than 0.2%Acidity (to phenolphthalein) ... Not more than I ml. of N. acid per 100 g.

... Not more than 0-01% Chloride ... ... Not more than 0.05% Sulphate ... ... Not more than 0.05%

The wool serge is treated at 90-95°c, for 15 min. in a relatively small volume of water containing the necessary amount of formic acid to give a pH value of the liquor of approx. 4. Cold water is added to bring the liquor ratio to 50:1 and the temperature to about 50°c. The required amount of D.A.N. dissolved in dilute ammonia (1 g. D.A.N., 9.5 ml. ammonia (sp.gr. 0.880) in 80 ml. hot water) is added during a period of 30 min. to the dye vessel while the cloth is moving through the liquor and at the same time formic acid is added to maintain the pH value of the dye liquor at about 4 (0.4 ml. of 90% formic acid in 80 ml. of cold water for each 0.5 ml. of 0.880 ammonia). The dyebath is raised to the boil in 45 min. and boiled for 90 min., when the cloth is hydroextracted without rinsing and dried.

It is not possible to obtain complete exhaustion, and to allow for this the amount of D.A.N. applied must be greater than the amount required on the standard. The amount actually present on the serge should be estimated by the following method—

### ESTIMATION OF D.A.N. ON TREATED SERGE

### Reagents

(1) Standard Solution—Dissolve 0.5 g, D.A.N. in warm M./60 trisodium phosphate (t.s.p.) solution (see below), cool, and make up to 1 litre with M./60 t.s.p. solution. Dilute 25 ml. of this solution to 250 ml. with M./60 t.s.p. solution. The solution obtained contains 0.05 mg. D.A.N. per ml.; and 5-, 10-, 15-, and 20-ml. aliquots, made up to 100 ml. with M./60 t.s.p. solution, give solutions uitable for measurement in an absorptiometer. The following readings, made on a Unicam S.P. 600 spectrophotometer at 435 mμ. using 1-cm. cells, are typical—

Conen. of D.A.N. (mg./100 ml.)	Optical Density
0.25	0.173
0.50	0.341
0.75	0.506
1.00	0.672

(2) M./60 Trisodium Phosphate Solution—Dissolve 19 g. of  ${\rm Na_3PO_4,12H_2O}$  in 3 litres of distilled water.

### Method

Immerse a shredded sample (0.5 g.) of the material in 20 ml. trisodium phosphate solution at 50-60°c, for 10 min. Decant the extract through a No. 2 sintered-glass funnel. Using forceps return to the bulk any fibres collected in the filter. Repeat this extraction procedure until the extract is no longer yellow in colour. Usually about six extractions are necessary.

Combine the extracts, make up a suitable volume with M./60 t.s.p. solution, e.g. 500 ml. or 1000 ml., and measure the D.A.N. content of the solution, preferably absorptiometrically, but alternatively by comparison with solutions of known strength in Nessler glasses or in a Duboscqtype colorimeter.

### Appendix 3

RECOMMENDED PROCEDURE FOR REARING AND HANDLING THE COMMON (WEBBING) CLOTHES MOTH Tineola bisselliella (Hum.)

### (1) Collection of Eggs

The following technique, which is flexible in detail, is based on the work of Heal<sup>2</sup>. Moths are

confined in a container with one or more sides of wire gauze having .7–8 meshes per centimetre (diameter of wire approx. 0.36 mm.), through which the moth lays its eggs on to a piece of undyed, scoured, lightly milled wool fabric which has been bound tightly round the gauze sides of the container. Gently detach the eggs from the cloth with a brush. The eggs must be free from mites.

### (2) Rearing Medium

The medium shall consist of undyed wool of mean fibre diameter less than  $24~\mu$ . in the form of dry combed noil\*, scoured yarn, scoured knitted fabric, or scoured cloth. The aqueous extract of the wool shall have a pH value within the range 7–9 when measured by the I.W.T.O. Standard Method <sup>1</sup>.

For impregnation use the procedure given below, except for noil, for which the cholesterol is to be omitted and appropriate modifications in manipulation are to be introduced to facilitate the retention of 10% of yeast. Impregnate scoured, undyed wool in the form of yarn or knitted or woven fabric with an aqueous dispersion of dried debittered brewer's yeast (Saccharomyces cerevisiae Hansen) and cholesterol of pharmaceutical quality (10 parts of yeast and 1 part of cholesterol by weight) so as to add to the wool approx. 10 parts of yeast and 1 part of cholesterol by weight.

The following method is suitable when only limited equipment is available—

For 200 g. of wool prepare 400 ml. of dispersion; grind 4 g. of cholesterol finely using a mortar and pestle; add in small quantities 40 g. of yeast, grinding thoroughly after each addition. Make a paste of this finely ground mixture with gradual additions of distilled water at 50–60°c. to a total volume of 400 ml.

Divide the wool to be treated into 20-g, portions. Pour 40 ml. of the well shaken dispersion into a padding trough or other suitable container and add a 20-g, portion of wool. Work the wool in the dispersion with the hands (clean rubber gloves can be worn if desired) to obtain thorough impregnation, and then pass the wool through squeeze rollers using a nip adjusted so as to allow the 20 g. of wool to retain 20 g. of dispersion.

The cloth or knitted fabric is passed through the squeeze rollers in open width. If squeeze rollers are not available, remove excess dispersion from the material by hand-squeezing as uniformly as possible.

Discard the dispersion left in the container; replace with 40 ml. of fresh dispersion and impregnate a further 20 g. of the wool as before. Proceed in this manner until all the wool has been treated.

If the division of the wool into 20-g. lots is inconvenient, any suitable weight can be used, provided that an appropriate volume of dispersion is used.

Dry, as quickly as possible, the supplemented wool at a temperature not exceeding 70°c. and condition by exposing for several days under the

<sup>&</sup>lt;sup>6</sup> Dry combed noil normally contains 0.5–1.0% of wool grease, which itself contains cholesterol.

test conditions of temperature and relative humidity.

(3) Rearing of Cultures

Distribute about 4,000 eggs upon 20 g. of the supplemented wool. Place the wool containing the eggs in a suitable ventilated container of volume about 2 litres, which is kept away from strong light. Maintain the temperature of the breeding room as described in § 6.2.

### Appendix 4

SUGGESTIONS FOR AN ACCEPTABLE LEVEL OF MOTHPROOFNESS

In its discussions on the interpretation of the term "mothproof", the Subcommittee has constantly borne in mind the fact that the majority of accepted commercial mothproofing treatments act as stomach poisons. The moth larvae have, therefore, to eat a small amount of fabric in order to acquire an injurious dose of the proofing agent, and in the test described in the Specification, where a relatively large number of larvae are confined

on a relatively small test pattern, some visual damage may be apparent at the end of the exposure period on patterns treated with concentrations of mothproofing agents which equal or surpass the acceptable level.

It is the considered opinion of the Subcommittee that a wool serge possesses a reasonable level of mothproofness if, when tested by this method, it contains the equivalent of 0.8% of D.A.N. It must be stressed that in practice a greater degree of mothproofness should be aimed at, so as to allow an adequate margin for inequalities in application and loss during use.

COMMERCE HOUSE

CHEAPSIDE

BRADFORD I YORKSHIRE

July 1956 (received 18th January 1957)

<sup>2</sup> Heal, R. E., J. Econ. Ent., 35, 249 (1942).

### References

<sup>1</sup> I.W.T.O., Method of Test for the Determination of the pH Value of a Water Extract of Wood (Bradford 1955).

### ERRATUM

A Study of the Photochemistry of Dyes on Proteins and other Substrates (J. W. Cumming, C. H. Giles, and A. E. McEachran, J.S.D.C., 72, 375 (Aug. 1956) — In Table II the names of the first two dyes in the first column should be transposed; i.e. data in the top line of the table refer to "Dimethyl-IV", and those in the second line to Cloth Red 2R (IV).

### Notes

# Meetings of Council and Committees March

Council-6th

Terms and Definitions-15th

Colour Index Editorial Panel-18th

Publications-19th

Review of Textile Progress-25th

Perkin Main-28th

Annual General Meeting-29th

Annual Meeting of Chairmen and Honorary Secretaries of Sections—29th

### Symposium 1957

### Recent Advances in the Colouring of Man-made Fibres

Arrangements for the Symposium to be held by the Society at the Palace Hotel, Buxton, during 18–20th September 1957 are well in hand. It is expected that the Symposium will have a markedly international character, as offers of papers have been received from as far afield as the U.S.A. and Japan in addition to Continental countries (France and Germany). It is hoped to include papers dealing with the application to viscose rayon, cellulose acetate, nylon, Terylene, and acrylic

fibres of acid, basic, and disperse dyes and also logwood.

### William Dunford Appel, M.S., F.S.D.C. Presentation of Memorial Medal by the A.S.T.M.

At the Sheraton-McAlpin Hotel in New York City on 21st March 1957 the Harold DeWitt Smith Memorial Medal was presented to Mr. W. D. Appel by Committee D-13 on Textiles of the American Society for Testing Materials. This medal is awarded at intervals of not less than one year for outstanding achievement in research on fibres and their utilisation. Mr. Appel is Chief of the Textiles Section and Assistant Chief of the Division of Organic and Fibrous Materials at the National Bureau of Standards in Washington, D.C., a former president of the AATCC, and a member of the AATCC Colour Index Editing Committee.

### Employment of Women as Technologists

In the daily press there has of late been much discussion on the desirability of more women being trained as scientists and technologists. In this connection it is interesting to note that the Honorary Secretaries of the West Riding Section

of the Society and of the Bradford and Leeds Junior Branches are all three women—Miss Esmée Smith, Mrs. B. Cope, and Mrs. J. M. Firth respectively.

### Perkin Centenary Presentation in Canada

The Canadian Association of Textile Colourists and Chemists has presented to the Department of Chemistry of McGill University, Montreal, Quebec, frames containing Mauve and fabric dyed with it, and also copies of Perkin's patents.

### Perkin Centenary Celebrations in India

The centenary of Perkin's discovery of Mauve was celebrated at the M.S. University of Baroda during 17-19th September 1956. The proceedings started with an introductory speech by Mr. D. Subba Rao, Dean of the Faculty of Technology and Engineering, in which he stressed Perkin's statement: "Not unto me, O Lord, not unto me, but unto Thy Great Name, let all the praise be". Dr. Suresh Sethna gave an account of Perkin's life, and Dr. R. L. Desai outlined the development of dyes from Mauve to the latest reactive dyes. Dr. R. D. Desai discussed, with the aid of tables of statistics, the present position of the Indian dye industry: the first factory started production in 1940, and now there are nine manufacturing firms and three more plants under construction; the range manufactured in India will include azo dyes and azoic components, Sulphur Black, and vat dyes. It is interesting to note that Indigo is now imported into India.

Following the talks, Mrs. Hansa Mehta, Vice-chancellor of the University, formally opened the centenary exhibition, which appears to have been similar to those held in this country (cf. J.S.D.C., 72, 566 (1956)). The exhibition, which was open to the public for two days, September 18th and 19th, is described in the November 1956 issue of the Indian Textile Journal, which contains also the papers by Dr. R. L. Desai and Dr. R. D. Desai (67, 82–89).

### Nederlandse Vereniging voor Textiel-Chemie

The new secretary of the Dutch society is H. Boezeman, and correspondence should be addressed to him at Dr. Schaepmanplantsoen 7, Helmond, Holland.

### Re-use of Water by Industry

An American symposium on this subject (Ind. Eng. Chem., 48, 2145–2171 (Dec. 1956)) emphasises the importance of the conservation of water, a problem which we will have to face even with our climate. Although none of the papers is directly concerned with the dyeing and finishing

industry, several might be studied with profit, e.g. those on the Re-use of Steam Condensate as Boiler Feed-water, Biological Fouling in Recirculating Cooling Water Systems, and Adaptation of Treated Sewage for Industrial Use.

### Legislation on Food Dyes

According to Chemistry and Industry (96, 26th January 1957), regulations concerning the colouring of food made by the following governments are abstracted in No. 4 and 5 of Current Food Additives Legislation (published by the F.A.O. in Rome see J.S.D.C., 72, 538 (1956)): New South Wales (food colours), Canada (colours), Denmark (tinned fruit and vegetables—permitted colours), Greece (fruit products (artificial colouring) including ice cream made from natural fruit juices), India (permitted food colours for fruit products), the Netherlands (permitted colours for margarine), New Zealand (permitted colours), South West Africa (standards for canned meat and fish products, including permitted colours; also methods of analysis), U.S.A. (colouring of the skins of oranges), and Yugoslavia (colours for food).

### Industrial Developments in Holland

The Royal Dutch Salt Industries Ltd. are to erect at Hengelo a plant for the production of hydrosulphite, most of the Dutch requirements for which are now imported. A new Anglo-Dutch company, Regout-Matthey Ltd., is to be founded to deal with the manufacture and marketing of pigments and "ceramic dyestuffs". A firm at The Hague, Lak-, Verf- & Inktfabriek "Premier", has developed a metal cleaning basin in which screen-printing frames can be readily cleaned with only a small quantity of solvent.

### **New Russian Textile Abstracts**

Beginning in January 1957 the All-Union Institute of Scientific and Technical Information is issuing two new series of "Express Information", viz. "Textile Industry" and "Artificial and Synthetic Materials". These new publications will contain abstracts of the most important work appearing in the foreign scientific and technical literature relating to the textile industry, and the abstracts may be illustrated by photographs, drawings, graphs, and tables. The two series of "Express Information" will have 48 issues a year, the price being 360 roubles.

### Ortalion-Italian 6-Nylon

The firm of Bemberg S.p.A. of Milan began production during 1956 of a new 6-nylon yarn, Ortalion, for which a plant has been built at Gozzano, Novara.

### New Books and Publications

Review of Textile Progress Volume VII 1955

Published jointly by the Textile Institute and the Society of Dyers and Colourists. Pp. 581. Price, 42s 0d. or \$6.00 (33s. 0d. to members of

the Institute or the Society).

Once again a team of about thirty authorities, drawn from industry, research associations, and educational establishments, have summarised a year's progress. In certain subjects, which have not been discussed for a year or two, e.g. chemistry of bast fibres, carpet production, felt, the review goes back as far as 1952. Sections of particular interest to most members of the Society will be those on colouring matters (K. Venkataraman), dyeing (F. V. Davis and C. B. Stevens), printing (A. Howarth), and finishing (C. A. Norris and R. G. Fargher). Foreign contributors have written the sections on colouring matters and the production of synthetic and cellulosic fibres.

Each section is largely self-contained; one wonders, however, whether this policy has not been taken too far, and whether stricter editorial control might not have prevented some overlapping. Duplication is sometimes necessary, but usually needless, as most readers will read related sections. The sections on Synthetic Fibres—Production and Synthetic Polymer Fibres—Chemistry overlap badly in places, as do those on Cellulose—

Chemistry and Fibre Physics.

The book is undoubtedly a "must" for anyone wishing to keep abreast of developments, and provides a most useful conspectus of the changing textile industry as a whole, whilst the printing and the format make it a pleasure to handle.

L. Peters

Sbianca, Tintura, Stampa, Finitura dei Tessili By G. Prelini 3rd edition, revised. 1956. Pp. xi + 454 + 122 illustrations in the text. Milan:

Ulrico Hoepli. Price, 3800 lire.

The state of development of the textile and ancillary industries of Italy is not widely known in this country, and therefore, apart from its own claims for attention, this book has proved a very

interesting one to review.

In the preface, the author claims that it has been written as an introduction, based on scientific principles, to the textile-processing industry for those who already possess a good general training in physics and chemistry. Apart from the very full bibliographies provided, which do include many references to fundamental studies, this claim is, perhaps, a little overexalted, for there is not much evidence in the text itself that processing is understood to be related at all closely to any very profound or modern systems of theoretical chemistry or physics. More utilitarian purposes are in fact revealed, for, at the beginning of the book, a scheme of studies in textile chemistry is reproduced, officially approved for use in technical institutions, and the reader is directed to the relevant pages for each item.

However, having settled the level, it must then be said that this book represents a very comprehensive treatment of the subject-matter of textile processing in all its practical aspects. It attempts to cover, more or less fully in accordance with their interest or importance, the properties of all the textile fibres as they affect processing, and to include most varieties of textile processes in some detail. The numerous illustrations are well selected, mostly to show typical processing machinery. In the dyeing and printing sections especially, there are competent treatments of the simple reactions and structural chemistry of dyes and other reagents, but the treatment is introductory and calls for no special mention.

The machinery illustrations are interesting when considered alone and away from the text. Occasionally they may be a little old fashioned, as when they include a picture of the Mettler constant-tension jigger of Sulzer, which, although very ingenious, must be regarded as of academic interest only, in a context where the evolution of jigger design is not being discussed. On the other hand, some of the information is up to the minute. There are good treatments of continuous bleaching and dyeing systems, high-temperature dyeing (with a better explanation of the action of the Barotor machine than can be found in most of the American articles on the subject), the new Zittau multinip roller printing machine, Vigoureux printing, mechanised screen printing, electrostatic flock printing, etc. Although they may, in some cases, be acting as agents or licensees for foreign firms, it is impressive to see what a large range of textileprocessing machinery the Italian firms can themselves provide. On the whole, the non-Italian machinery represented is of German origin, and the native designs, where they are not original, tend to follow German patterns.

The text, which, for a book of this kind, seems to have no notable omissions, is well arranged, and the explanations, although a little oversimplified on occasion, are clear and direct. They read like a rather attractive course of lectures extending over a number of sessions. This book should, in fact, prove very helpful to teachers of textile technology, who should not let themselves be intimidated by the language difficulty, because technical Italian of this kind is relatively easy, and a little hard work with the dictionary during the first chapter can enable one to surprise oneself.

The very long bibliography given in the front of the book may prove a little confusing to the readers for whom it is intended. The select list of textbooks which it includes is quite good and names a number of well known works. A further list of references to published articles and papers is so large, and ranges over so long a period, that it must be difficult for the beginner to decide which is the most important or which will help him the most. A good deal of the earlier material has now found its way into one or other of the books cited, and has probably been dealt with there in sufficient detail for most of the potential readers. These references would be more significant if they were weeded out a little and then introduced into the

text at the appropriate places.

The printing and the paper are satisfactory. The binding is not very strong and should be regarded as temporary only. On the glazed paper cover is a lively design in montage, made up from reproductions of textile prints, so that the whole presentation is different from that to which we have become accustomed for the textbooks current here.

H. A. Turner

### Maschinen und Technologie der Nassveredlung von Zellulosefasertextilien

By A. Grünert. Pp. 336 with 333 illustrations in the text. Berlin: VEB Verlag Technik. 1955. Price. DM 32 00.

The contents of this modest-looking book are extremely concentrated, and it represents perhaps the most competent survey of textile-processing machinery that has appeared for many years. There has been occasion in recent times to comment on the somewhat parochial attitude of some German wrifers on textile-chemical subjects, and this book might be considered to be a rather striking example of the same tendency, since more than half the illustrations are of machinery made by Zittau, and a very large proportion of the rest come also from German sources. In this country. we tend to react rather strongly against treatments which pretend to be balanced and inclusive and yet seem to "plug" particular interests. After all, inventiveness and industry are not confined to one region of the earth. In this case, however, the reaction must be greatly tempered by the thought that the chief author (four other collaborators are mentioned on the title page) is giving us the benefit of his long experience in a particular milieu and with very specific preoccupations. There is no doubt that he is a sound guide on the whole, and the team has gone to great trouble to collect, and present logically, details of certain branches of the subject that are not readily accessible in so complete a form elsewhere. A good example is the section which deals with the equipment of screen printing, both hand and mechanised.

The sign of a good book is generally that, out of all the details that are necessary for completeness, a clear philosophy should be seen to take form. By this criterion, the book is indeed good, for it does reflect the versatility of the textile machinery industry and its adaptability to the changing requirements of processing, brought about by change in scale, change in package or other form of presentation of the textile material, change in the chemical properties of this material, and so on. Against this is also a conservatism of design, so that revolutionary transformations are comparatively few. This last feature may be due to the fact that practically all textile machinery is designed in the drawing office. The time when the chemical engineer will intervene much more decisively cannot be long delayed, so that the present book may be one of the last of its kind.

The flavour of the work may best be given by quoting some of the features that impress on a first reading. As might be expected with a German book, the section on machinery for treating varn is very well done. The whole subject of impregnation mangles, and mangle-developing sets, is very informative, and gives examples of a large number of designs where the contact path of cloth with liquor is made as long as possible, while the volume is kept low. The transition stages between mangle and jigger are also particularly well illustrated, and emphasise some of the empirical devices that seem to have been developed in advance to take advantage of the lessons on liquor interchange between cloth and bath in the common jigger, recently discussed by Ashworth of Courtaulds Ltd. The book must have been in preparation just a little too early for the Küsters mangle to be included. The sections on printing are, as indicated already, very full and very interesting, and, along with an extended treatment of the many mechanical devices on the printing machines proper, reflect the German fondness for large steamers, and hot-flues with intricate types of spiral cloth path. Apart from its applications in mercerising, the stenter appears to be treated almost exclusively as a drying machine, and in this way a consideration of the many forms of guiding mechanism, especially for the pin-stenter, is almost absent. Perhaps a little more could also have been said about tensionless drying machines of the "air-lay" and similar types.

In this, as in other books on textile processing, there is revealed one branch in which German design seems to be unprogressive, and that is in burner systems on singeing machines for piecegoods. All the secondary appurtenances may be quite elaborate, but there seems to have been no move from the plain strip burner applied without artificial prolongation of the time of contact between the cloth and the hot gases of the flame. This is surprising in view of very high singeing speeds recently quoted by Piepers and also because Haubold did, some years ago, start the idea of looping the cloth and putting a flame at the mouth of the loop, an idea much improved in the Parex system. It would be interesting to hear more on

this subject.

There are one or two items that one would wish to have seen treated in the same competent manner as those which have received the special attention of the writers, but to demand very much more from a book of this size would be unreasonable. It is one which all interested in the design and the operation of textile-processing machinery cannot afford to miss.

H. A. Turner

### Reports on the Progress of Applied Chemistry Volume XL 1955

London: Society of Chemical Industry. [1957.] Pp. 1000. Price, 40s. 0d. (20s. 0d. to S.C.I. . members).

Owing to the printers' strike last year the publication of this annual report was slightly delayed. It records new developments and continued progress in all branches of chemical industry.

H. E. Nursten (University of Leeds) reviews developments in intermediates and anthraquinone, phthalocyanine, sulphur, indigoid, and other dyes, and also fluorescent brightening agents.

In his review on textile finishing A. J. Hall mentions the improved bleaching processes for nylon by treatment with permonosulphuric acid under strongly acid conditions, and for polyacrylonitrile by a two-stage process using a hot potassium permanganate solution and then a boiling solution of sodium bisulphite. Reference is made to BP 738,014 of the Distillers Co. Ltd. recommending n-butanol for the solvent scouring of raw wool. The section on dveing and printing describes continuous dyeing processes for cotton and other cellulosic fibres, and recent technical developments on the Standfast dyeing machine. There is also a description of the Pad-Roll method devised in Sweden, of the methods of dveing cellulose triacetate, and of suitable carriers for dveing synthetic fibres. Progress in textile finishing has been largely connected with the use of synthetic resins to secure crease-resistance, dimensional stability, and increased permanency of mechanical finishes. There are 144 references to publications and patents relating to textile finishing.

R. J. E. Cumberbirch writes on the reactivity of cellulose and present theoretical views on the structure of cellulose. J. F. Keggin on protein fibres mentions the important advances which have been made during the year in the analysis of proteins by using the techniques of chromatography and end-group analysis. He also surveys recent developments in the spinning of regenerated

protein fibres.

In the section on synthetic fibres, various authors survey the progress in polyamide, polyester, and acrylic fibres. The acrylics are becoming increasingly important: Orlon (DuP) production has increased by 30%, and it is reported that 75% of American sweater production is of acrylic materials. Acrilan (Chemstrand) is also developing satisfactorily, and apart from Chemstrand's plans to produce Acrilan in the United Kingdom, British firms seem to have intentions of producing similar fibres. It was announced that another acrylic fibre, Prelana, is produced in East Germany, and it is claimed to resemble, and to have superior

qualities to, merino wool.

In the chapter on fats, oils, and detergents K. Burrow (Thomas Hedley & Co. Ltd.) reports that in the U.S.A. synthetic detergents accounted for 58% of the total U.S. detergent market in 1955 (compared with 53% in 1954) and it is estimated that by 1962 they will account for 75% of the total detergent sales. In Britain synthetic detergents now account for 40% of the total detergent sales. Dodecylbenzene continues to be by far the most widely used organic raw material for synthetic detergents, the alkylarylsulphonates representing 51% of the total. There is a survey of developments in manufacturing processes, additives, spray-drying techniques, and in analytical testing and evaluation methods. A. Carruthers (British Sugar Corpn. Research Laboratories), in reviewing developments in sugar, mentions the research

studies on the esterification of sugar to produce a detergent; such sucro esters (Hass, L. B., Sugar, 50, 43 (1955)) are reported to be comparable in their properties with dodecylbenzenesulphonate detergents. Further studies on sugar-based detergents have since been published by Snell (Soap and Chemical Specialties, Dec. 1956, 47).

Other contributors review the progress made in inorganic chemistry, fuel and fuel products, metals, rubber, leather, antibiotics, fine chemicals and medicinal substances, photographic materials and processes, cosmetic and toilet preparations, plastics, paints, chemical engineering, food and

agriculture, etc.

Anson Quinton (Queen Elizabeth Hospital. Birmingham) writes on industrial uses of isotopes, a subject which the non-specialist also will find interesting. The widespread use of radio-isotopes in industry dates from 1950, when they became available in appreciable quantities from the atomic energy authorities. At present there are 28 radioactive isotopes available for use in industry, and their industrial use will continue to increase, e.g. for the elimination of static electricity in textiles and plastics, and for thickness and moisture control in papermaking; isotope techniques are also applicable to wear and corrosion tests. Many industries, like the dairy industry, depend on the separation of solid and liquid phases, and by making the liquid phase radioactive, the efficiency of separation can be determined. The risk of contamination of surfaces by radicactive substances must not be overlooked, and radiation risks and hazards in the scientific and industrial use of atomic energy have been explained by Spiers (Chem. and Ind., 1446 (1955) ).

The book is well printed and produced, with well compiled indexes and references. It is an invaluable

source of information and knowledge.

A. F. Kertess

Irradiation Colours and Luminescence A Contribution to Mineral Physics

By Karl Przibram, translated and revised by John E. Caffyn. Pp. xiv + 332. London; Pergamon Press Ltd. 1956, Price, 63s. 0d.

This book deals with a subject which lies between the interests of the mineralogist and those of the physicist but is probably nearer to the former. The current theory of the coloration of minerals as being due to radiation, and the importance of lattice imperfections in coloration, are discussed in the general section of the book. This is supplemented by detailed sections dealing with the more important types of mineral. The inclusion of an extensive bibliography makes this a valuable source book, but the nature of the subject-matter is such that it is not particularly attractive to the casual reader.

E. ATHERTON

Modern Instruments in Chemical Analysis By F. M. Biffen and W. Seaman. Pp. ix + 333. New York: McGraw-Hill Book Co. Inc. 1956, Price. \$7.50.

Increasing use is made of instrumental methods of analysis in chemical laboratories of all kinds. Such methods are rapid, require only small amounts of material, and may often be used by comparatively unskilled staff. The aim of this book is to present to the general analytical chemist information regarding a number of experimental techniques and equipment which will help in the choice of the most direct and economical method for a particular analysis.

Following an introduction in which the choice of method, the sample, and its preparation are briefly considered, there are seven chapters dealing with methods depending on measurements of electromagnetic radiations. A chapter on emission spectroscopy is followed by one on flame photometry. Another on visual and ultraviolet spectroscopy should be of interest to readers of this Journal, as some of the methods of analysis given are applicable to dyes. Colorimetric analysis is, however, only briefly considered, and absorptiometers of the Spekker and similar types are not considered in detail. Turbidimetric and fluorimetric analyses do not seem to be considered. There are brief chapters on X-ray diffraction and on infrared, Raman, and mass spectroscopy. Five chapters devoted to electrochemical methods comprise a useful survey of such methods and accounts of polarography and of potentiometric, conductimetric, and coulometric analysis. A final chapter deals with radioactivity in analysis.

Each chapter gives brief outlines of the theory of the method and the instruments used and some typical applications. In a book of this type it is essential that the scope and the limitations of each method be clearly shown and sufficient theory and description given to provide the reader with at least the minimum necessary to follow the working of the method and the applications of the technique to analysis. These requirements are generally met, although the theory provided is often barely the minimum required. Each chapter is, however, supplemented by a bibliography and list of references. Diagrams illustrating principles are clear, but some of the photographs are not particularly so and often illustrate apparatus not generally available in this country. The book does. however, provide a good deal of information, in a readable yet accurate form, likely to be of value to chemists in general and analysts in particular.

W. R. MOORE

### The Chemical Industry in Europe

Organisation for European Economic Co-operation.
Pp. 211. Paris. 1956. Price, 14s. 0d. (\$2.50).
This study constitutes the third annual report on the chemical industry drawn up by the Chemical Products Committee of O.E.E.C. The chemical industry represents about 3% of the gross national product and employs about 1·2% of the total labour force. The general trend of dye production was to become stabilised at rather lower levels during 1955 after the great increase in output in 1954. The fall in value was rather greater than the fall in output, owing to keen competition forcing down prices. In general, productive capacity was not fully utilised, and in view of difficulties facing the textile industry, outlets for dyes in other industries,

such as paper and plastics, may offer better prospects. World statistics show that dye production in other countries continued to increase, and European exporters may expect to meet increased competition from the U.S.A. and, more recently, the U.S.S.R.

The detailed tables of statistics and the reviews of the situation in different branches and in different countries make this study a valuable source of information on the economic aspects of the chemical industry in Europe. C.J.W.H.

### Synthetic Organic Chemicals United States Production and Sales 1955

United States Tariff Commission, Report No. 198, Second Series. Pp. viii + 187. Washington: United States Government Printing Office. 1956. Price, 65 cents.

This thirty-ninth annual report is a valuable source of statistics relating to dyes and intermediates in the U.S.A. Compared with 1954, American production of dyes increased in 1955 by 17.5% to 168 million lb., and that of toners and lakes by 11.3% to 45 million lb. Data are given for intermediates and also for dyes listed by (old) Colour Index numbers, the C.I. dyes accounting for 70.8% of the output of all dyes. Production and sales of dyes are grouped also by chemical class and by application class. Part III of the report lists the manufacturers of the different dyes, and in an appendix are tabulated the imports into the U.S.A. of the different classes of dyes for the years 1953 - 1955. C.J.W.H.

## ASTM Standards on Textile Materials (with related information)

Prepared by ASTM Committee D-13 on Textile Materials. Pp. xxii + 814. Philadelphia: American Society for Testing Materials. November 1956. Price, \$6.25 (\$4.70 to A.S.T.M. members).

The latest A.S.T.M. tentative and standard methods of tests, specifications, and definitions pertaining to textile materials are included in this book. Of the 126 standards and tentatives, 21 are new and 8 have been revised since the previous edition of the book was published in January 1956. Fourteen appendixes cover 13 proposed recommended practices, methods of test, and specifications as well as a table of the basic properties of textile fibres and a psychrometric table for relative humidity.

The standards cover inter alia terms and definitions, testing machines, humidity testing, interlaboratory testing, identification for qualitative analysis, quantitative analysis, resistance to insect pests and microorganisms, hosiery, asbestos textiles, bast and leaf fibre textiles, glass textiles, warp-knit fabrics, pile fabrics, and non-woven fabrics.

C.J.W.H.

### Paper Terminology A Glossary of Technical Terms

Compiled and published by Spalding & Hodge Ltd.
Pp. 74. London: Revised 1954. No price.
This handy little booklet, issued by an old established firm of papermakers, will probably be

of use especially to those who are less directly concerned with the paper trade. Appendixes give extracts from paper trade customs and lists of paper sizes. A rather long preface ends with a statement of the very commendable aim: "to win the confidence and goodwill of those with whom we come in contact, to deal pleasantly with them, and to make it a felicity to do business with us."

to make it a felicity to do business with us."

The glossary itself makes not uninteresting reading, and one's impression is that paper terminology entails the use of everyday words in very special senses rather than the employment of any large number of unusual words. In several cases information rather than what might strictly be regarded as definitions is given, e.g. the use of aniline sulphate to detect the presence of esparto in paper, and the manufacture of the paper for Bank of England notes from new unbleached linen. Carpet brown is imitation felt manufactured from waste fibre and used for underlaying carpets, and decalcomania (from the French décalquer), a word which troubles some readers of our abstracts, is a transfer paper for conveying a design to pottery, etc. Under durability are given the specifications laid down by the Library Association for two main grades of durable paper. Half stuff can be beaten whole", and long elephant is used in the wallpaper trade. In view of the difficulties encountered in "tinctorial terminology", it is interesting to note

the definitions of smalts— "a blue, light-fast pigment, consisting of ground cobalt silicate. Used as a dye in making the best qualities of azure writing and ledger papers"— and of staining— "a method of colouring a paper by treating it in the web with a solution of a dyestuff, as an afterprocess."

C.J.W.H.

### New Books received

- ASTM Standards on Textile Materials. Philadelphia; American Society for Testing Materials. Nov. 1956, Pp. xxii + 814. \$6.25.
- Hydrogen Ions— Their Determination and Importance in Pure and Industrial Chemistry. Volume I. Hubert T. S. BRITTON, London: Chapman & Hall Ltd. 4th edition 1955. Pp. xix + 476. 75s. 0d.
- Chimie des Peintures Vernis et Pigments. G. Champetter, H. Rabaté, and J. L. Rabaté. Paris: Dunod. 1956, Volume I Pp. viii + 661. Volume II Pp. vi + 643.
- Semimicro Qualitative Organic Analysis— The Systematic Identification of Organic Compounds. Nicholas D, Cherosus and John B. Entrices. New York: Interscience Publishers Inc. 2nd edition 1957. Pp. xiv + 774. \$9.00.
- Proceedings of the International Wool Textile Research Conference, Australia 1955, Volume F. Melbourne: Commonwealth Scientific and Industrial Research Organisation, 1956, Pp. 228,
- Year Book of the Textile Institute. No. 9 (1956-57). Manchester: Textile Institute. Pp. 66 + 206, 15s. 0d, to non-members.
- Polymer Solutions. H. Tompa. London: Butterworths Scientific Publications. 1956. Pp. xiv + 325, 55s. 0d.

### Manufacturers' Publications and Pattern Cards

The Society does not accept any responsibility with regard to the statements in the following notes.

Any publication abstracted may be referred to by members of the Society on application to Dr. C. B. Stevens, Dyeing Department, Leeds University

### Badische Anilin- und Soda-Fabrik AG.

Basolan Chrome Olive GGM—This homogeneous chrome dye can be applied by both the afterchrome and single-bath methods to give dyeings of good fastness to light and wet treatments on all forms of wool. It is also suitable for dyeing polyamide fibres by the same two methods and is of interest on silk. Fastness figures for an afterchrome dyeing on wool include—Light 5, alkaline milling b 4–5, potting 3–4, carbonising a (sulphuric acid-neutralised) 4–5.

### Farbenfabriken Bayer AG.

THE ACRAMIN FKLN PROCESS FOR THE DYEING OF PIECE GOODS—This card contains pad dyeings on cotton cloth of a range of 16 resin-bonded pigments using Acramin FKLN as binding agent. Acramin FKLN is a new product superior in binding power to Acramin FKL, used previously. Dyeings retain a higher degree of lustre and are faster to mechanical treatments, while the binder is unaffected by prolonged exposure to sunlight. Persoftal FKL is still recommended as a softening agent and ammonium nitrate, sulphate, or chloride or diammonium phosphate as acid-forming catalyst.

ammonium phosphate as acid-forming catalyst.

ACRAMIN SL— This is a new resin-forming composition, which contains all the necessary ingredients for producing stable films and which in conjunction with the Acramin F dyes gives pigment prints of high brilliance and soft textile fibres, especially cotton, regenerated cellulose rayon, cellulose acetate, polyamides, wool, and silk. The preparation of print pastes is extremely simple, and heir flow properties, in both machine and screen printing, are excellent. Prints on cotton cambric and spun rayon muslin of 17 Acramin F dyes are included.

ASTRAZON BLUE 6GLL— This dye is primarily intended

ASTRAZON BLUE 6GLL—This dye is primarily intended for direct printing on cellulose acetate and polyacrylonitrile fibres, on which it gives very bright greenish blues of very good fastness to light, even in pale depths. It may be printed alongside disperse dyes as well as other members of the Astrazon range, but should not be used as a component in mixtures with the latter. Fastness figures for a print on polyscrylonitrile cloth include—Light (§ standard depth) 7, washing b (140°r.) 2, chlorine b 4.

Astrazon Yellow 6GL—This basic dye is recom-

ASTRAZON YELLOW 6GL—This basic dye is recommended for dyeing and printing polyacrylonitrile fibres and also for printing cellulose acetate. It gives bright greenish yellows of very good fastness to light and wet treatments. It has high affinity for polyacrylonitrile fibres of widely differing origin and may be used in mixtures with other members of the range and also the Astra dyes. Normally dyed at the boil in presence of acetic acid and Avolan A, it can also be applied at 105–108°c. Fastness figures for a dyeing on polyacrylonitrile include—Light 6-7, washing b (140°r.) 5, perspiration 5, milling b (severe milling) 5.

ECLAN WA EXTRA CONC.—This is the liquid form of Eulan WA highly conc., and is applied in the same manner as the latter during the final wet process in the finishing of wool and mixtures containing wool to render them moth-proof. Used in sufficient amount (73% on the dry weight of the goods) it also inhibits the growth of bacteria, although it is not a bactericide. Repeated washing removes it, so it is primarily intended for application to materials which are not normally laundered, in which case permanent protection is given. The storage life of the liquid is 3-4 woother.

Levapon WNI—A non-ionic surface-active agent primarily intended for wool scouring.

BLANKOPHOR ACF—A fluorescent brightening agent for use on polyester and polyacrylonitrile fibres and also on secondary cellulose acetate and cellulose triacetate. It is resistant to peroxide and sodium chlorate and is thus suitable for application during bleaching. The brightening effect has good fastness to wet treatments.

SIRIUS SUPRA GREEN GL- This direct dye gives full greens of good fastness to light on natural and regenerated cellulosic fibres. Aftertreatment of dyeir gs with Levogen WW or FW considerably improves their fastness to wet treatments with little, if any, reduction in light fastness. It is also very suitable for crease-resist finishing, since in this case also no significant change in the bue or the light fastness of the dyeings occurs. It is also of interest on silk or cotton-viscose rayon mixtures, on which solid dyeings are obtained. Using Type 8000, cellulose acetate is reserved. Fastness figures for a dyeing on cotton aftertreated with Levogen W include Light 5-6, washing

a (104°F.) 4, perspiration 5.
SUPRANOL BRILLIANT RED 6BW—This acid dye gives bluish reds on wool and is particularly suitable for dyeing slubbing and knitting yarns, particularly for sportswear and swim suits. It is also of interest on silk, and its high affinity from neutral baths makes it suitable for dyeing the wool or silk component of mixtures. Fastness figures on wool include— Light 4-5, washing b (140°r.) 4, perspiration 5, sea water 5.

### Farbwerke Hoechst AG.

OFNA-PERL SALT BBA- This is a stabilised diazo compound which may be applied to polyamide fibres together with appropriate Naphtols, the insoluble azo compound being produced inside the fibre by treating the "dyed" materials in a warm, dilute sulphuric scid solution. The colourings obtained, which include dark blues, violets, blue-greens, and greys, have very good fastness to light and wet treatments. Mixtures of polyamide fibres and cellulosic fibres may be dyed in two stages, the polyamide portion being dyed by incorporating Ofna-perl Salt RRA in the naphtholating bath, and then the azoic dye being produced on the cellulosic component by treating the material with the appropriate Fast Salt.

OFNA-PERL SALT RRA-This is similar in properties and uses to the BBA brand (it was actually the first of this type of product to be introduced). It gives reds, yellows, browns, and violets on polyamide materials, including loose fibre, sliver, yarn, and cloth. It is also of interest for dyeing mixtures of polyamide fibres with cellulosic fibres and wool, by using Naphtols and/or Ofna-lan RR and Ofna-lan Base REA and RGE in conjunction with it in the same operation. Dyeings are included of 15 combinations on filament Perlon, one on staple Perlon, two on 80:20 wool-Perlon yarn, one on 70:30 regenerated cellulosic rayon staple-Perlon yarn, and one on 50:5 cotton-Perlon yarn.

OFNA-PERL SALT SBR- This further addition to the Ofna-perl range of stabilised diazo compounds is applied to polyamide fibres in conjunction with suitable Naphtols in the same way as the RRA and BBA brands to give greys and blacks, four examples of which are shown on Helanca crimped nylon and three on Perlon staple yarn.

INDANTEREN BRILLIANT GREEN H3G- This homogeneous vat dye gives very bright bluish greens when dyed or printed on cellulosic materials. It is particularly suitable for use on curtain and furnishing materials, and materials dyed to any depth may be marked with the Indanthren fastness label. A Type No. 8059 is available for pigmentation methods of application. It is of interest on silk for fast-to-washing styles but not for curtains and hangings. Light to medium-depth dyeings on cellulosic fibres are readily dischargeable to white. Fastness figures on cotton include-Light 7, washing (c) 4-5, chlorine (a, b) 4-5.

NAPETOL AS-GP AND AS-RP-These two azoic coupling components are designed for use with the special stabilised diazo component Ofna-perl Salt SBR for producing browns of satisfactory fastness to light and very good fastness to washing on polyamide fibres. In combination with Naphtols AS-SG, AS-SR, and AS-LB a wide range of stocking colours from brown to olive-grey may be produced. Twelve such colours are illustrated on crimped nylon yarn, and three more on staple Perlon yarn.

### Imperial Chemical Industries Ltd

Lissapol D-This new detergent, described in Auxiliary Products Pamphlet No. 102, is essentially the sodium salt of cetyl oleyl sulphate and is marketed in both paste and powder forms. It is an efficient detergent for all

fibres and is also of particular interest as a dyebath addition, e.g. when dyeing Torylene polyester fibre. Typical applications in the textile, paper, fur, and leather industries are described.

Vantoc AL—An aqueous composition containing 10% of higher-alkyltrimethylammonium bromides possessing marked bactericidal properties and devised for use in industries concerned with the production, processing, and

distribution of food and drink.

VANTOC N POWDER— As Vantoc AL, but containing over 90% of active agent in powder form.

VANTROPOL BQ — An aqueous solution of a mixture of a non-ionic detergent and a quaternary ammonium type of bactericide for use in catering establishments.

STUDIES OF THE DYEING OF NYLON WITH ACID DYES-

The Dynam of Allos with Acts Dress A reprint of a paper by E. Atherton and R. H. Peters (Text. Research J., 26, 497 (July 1956)).

ORGANIC POLYMOCYANATES—THEIR USE IN THE SURFACE-COATINGS INDUSTRY—A reprint of a paper by H. A. Hampton and R. Hurd (J. Oil & Col. Chem. Assocn., 39, 609 (Aug. 1956)).

Technical Information Leaflets—Dyehouse No. 335, Procion Brilliant Red 2BS: Storage of Dyed or Printed Materials -- It has been found that deep dyeings or prints of Procion Brilliant Red 2B on cotton may stain adjacent white material after storage in a humid, acid atmosphere followed by saturation with water. effect is much less marked with viscose rayon and with pale to medium colourings on cotton or with resin-finished materials. The effect is due to migration of the very small amount of non-substantive dye which is liberated by breakdown of the dye-fibre linkage under these conditions, and the goods may be restored to their original condition by mild washing.

Dychouse No. 336. The Dycing of Cellulose Triacetate: Supplement No. 4. The Use of a Carrier for the Production of Black Shades - A short process is described for producing a black at 90-100°c, using Brenthol OT and Dispersol Diazo Black B in presence of diethyl phthalate as carrier. At 100°c., 6% of the carrier is sufficient, but 10% is required at 90°c. Details are also given for producing a black on a cellulose triacetate-viscose rayon mixture. two-bath process is generally necessary to avoid the change in hue of the direct dye during diazotisation, but Chlorazol Black LF can be applied from the same bath as the Brenthol OT and Dispersol Diazo Black B.

Dychouse No. 337. The Effect of the Crease-resist Process
in Disperse Dyes applied to Triacetate-Viscose Rayon Blends - Fastness figures are given for the change in hue and the staining of adjacent white material during a pleating process—10 min. at 1.06 kg,/cm.<sup>2</sup> (15 lb,/sq.in.) using dry steam—carried out on dyeings of a range of disperse dyes. It was observed that only with Duranol Violets 2R and RN was there a severe change in hue resulting from the crease-resist treatment, but severe changes in hue do occur during subsequent pleating, particularly if intermediate washing-off, prior to pleating, is omitted.

Dychouse No. 338. Methanol Copying Blue BS; Observations arising from Manufacturing Experience,

Blacks in Fall-on Effects using Dychouse No. 339. Alcian X Dyes - Details are given of the most suitable types of black and methods of application for producing fast outline blacks in designs where an Alcian X dye, particularly Blue 8EX, subsequently falls on the black component.

Dychouse No. 340. 1957 Spring and Summer Color Card

of America: Man-made Fibres and Silk.
Dychouse No. 341. The Dycing of "Terylene" Polyester
Fibre: Supplement No. 7. Modification of Scouring and
Clearing Recommendations—Small amounts of caustic soda carried over from scouring can cause fibre degradation during subsequent dyeing at 130°c.; soda ash is therefore recommended for scouring followed by rinsing in water containing I part of acetic acid (30%) per 1000 parts of water. Similarly, excessive "reduction clearing" cause damage, particularly in presence of Lissolamine A. It is now recommended that the temperature be reduced from 60-70°c. to 45-50°c. Alternatively, Lissolamine A can be replaced by Dispersol VL. Since, however, the latter is not as effective as a clearing agent, it is recommended that precautions should be taken during dyeing, e.g. by adding dye in portions, to reduce the need for severe clearing conditions. The risk is greatest with loose fibre and slubbing; yarn and, especially, cloth may be cleared

at 60°c. in presence of Lissolamine A if required.

Dyehouse No. 342. The Dyeing of Terylene Polyester
Fibre Ladies' Hose— The recently introduced 25-denier, 3-filament hose may be dyed the current fashion colours using disperse dyes at 100°c, without a carrier. Residual surface dye must be removed, if necessary by a "reduction clear", to avoid low fastness and prevent a change in hue during postboarding.

Dychouse No. 343. Lissapol GL Paste in the Laundry: Medium Temperature Process for White Work.

Dyehouse No. 345. Lissatan AC: Pitch Control in the Paper Industry.

Dyehouse No. 347. Pigmentation of Polyvinyl Acetate Emulsion Paints.

Dyehouse No. 349 (replaces No. 237). The Dyeing of "Terylene" Polyester Fibre: Supplement No. 2. A New Process for Blacks on Loose Staple, Slubbing, and Yarnmodifications include precautions to prevent degradation by caustic alkali during scouring, dyeing, and "reduction-clearing", and a note on the importance of avoiding excess of Dispersol Diazo Black B, which can cause a reduction in fastness to sublimation.

Rubber No. 43. Nitrile Rubbers having Low Compression Set at High Temperatures,

DURAZOL GREEN BL- This direct dye gives bluish greens of good fastness to light on cotton and viscose rayon and is particularly suitable for use on dress materials, velvets, and carpeting and, in full depths, for dyeing filament viscose rayon for furnishings. It is also of interest on silk. It is not recommended for dyeing rayon subsequently to be crease-resisted, nor does it cover irregularly dyeing viscose. It is suitable but of only limited interest for direct printing; grounds cannot be discharged to a satisfactory white but may be of some interest for coloured discharges. Fastness figures on viscose rayon include Light 5-6, washing (Test No. 2, once at 55°c.) 3, perspiration 4-5.

DURANOL PRINTING BLACK R- This highly concentrated disperse dye is marketed in paste form specially standardised for printing. It gives dense blacks on cellulose acetate and nylon. It does not sublime during normal steaming processes for these materials and with-stands "crushing" during roller printing. It is of no during roller printing. It is of no interest for printing cellulose triacetate or Terylene polyester fibre, and is neither dischargeable nor dischargeresistant. Fastness figures on cellulose acetate include-Light 5, washing (Test No. 1, once at 40°c.) 3, perspiration

Water-soluble Taionol Dyes: M Brands—This card describes a range of 12 water-soluble sulphur dyes. Dyeings in two depths on unbleached cotton cloth are shown together with copper-chrome-aftertreated dyeings where this treatment is applicable. It includes Thionol Sky Blue 6BM and Navy Blue CM, added to the range since the first edition. For dyeing cotton yarn packages it is now advised that, in order to promote level dyeing, salt should be added as a solution and in two or three equal portions, care being taken to keep the salt concentration within the limits prescribed. An additional note describes the application of these dyes to viscose rayon staple, yarn, and cloth, on which they are of particular interest where the colour and the fastness afforded by sulphur dyes are adequate.

SUBJECT INDEX TO RECENT DYESTUFFS DIVISION Technical Publications: 3rd Edition. January 1945. TO JANUARY 1946.

### VEB Farbenfabriken Wolfen

DYES AND AUXILIARIES FOR COLOURING PAPER - This pattern card (German text only) describes auxiliary products and dyes for colouring paper and contains dyeings in two depths of 10 acid dyes, 31 direct dyes (including 12 fast-to-light types), 3 basic dyes, and 2 pigments.

FAST-TO-LIGHT DIRECT DYES FOR WRITING PAPER-This card (German text only) contains 9 pastel colourings including one mixture.

SUDAN DYES FOR CANDLES, SHOE CREAMS, WAX Polishes, Oils, Fats, and Lakes—Fourteen examples are included in this card (German text only).

DIRECT AND ACID DYES FOR POLYAMIDE MOULDINGS-The use of these is illustrated by a range of 26 colourings on polyamide buttons.

### Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., 68, 23 (Jan. 1952), and also, together with symbols and the periodicals abstracted, in the annual index

### I-PLANT; MACHINERY; BUILDINGS

Continuous Washing and Aftertreatment of Parallel Threads of Regenerated Cellulose

Vereinigte Glanzstoff-Fabriken

BP 764,115

BP 765,656 Cloth Guide for Fabric entering Drying or other

Textile-finishing Machine Allmänna Svenska Elektriska BP 764.133

Nylon-preboarding Machine

E. E. Bellmann, R. Kronsbein, and F. Bellmann

BP 764,739 A vertical, cylindrical retort has apertures to receive, alternately from opposite sides, carriages on which hose forms are vertically mounted. Economy in steam space is obtained by mounting doors and filling pieces on a rotatable plate within the retort. The filling pieces are also used to exert pressure on the closed doors.

Garment-dyeing, Washing, or Dry-cleaning Machines Cherry Tree Machine Co.

A drum-type machine whose construction and the arrangement of the settling tank permit any desired degree of "dip" without affecting the automatic removal of scum from the liquor. In addition there is no need for a pump or any balancing of the circulation system.

Treating Paper during Manufacture

Combined Locks Paper Co.

In order to prevent adhesion between a travelling web, particularly of light-weight papers, and the press rolls and consequent lapping of the web, moisture is expressed from the web, supported on a felt, by a first press roll, over which the felt passes, and a second roll having a roughened surface for levelling the paper and for even expression of water. A moistening, soft-rubber-covered roll is in contact with the second roll to form a nip, in which a sufficient pocl of water is maintained to carry away fibre fragments, so that a film of water is deposited on the second roll to prevent the web from sticking to it.

Paper-coating Apparatus

Champion Paper & Fibre Co. BP 759,281
In the machine-coating process of BP 499,532, slack portions of the web may receive excessive coating weights, which may cause fouling of drying cylinders. These difficulties can be eliminated by means of a doctor consisting of a flexible cylindrical doctor rod, a flexible socket in which the rod is rotatably mounted, and a spring support for the socket which yields when the pressure of the web is too strong and springs out to maintain proper pressure on slack portions of the web. A resilient carrier (rubbercovered rolls or endless belt) may be provided which supports the web against the pressure of the doctor.

Laboratory Dyeing Machine (XIV p. 187)

### II— WATER AND EFFLUENTS

Soaps and Sequestering Agents-Complete Detergents M. R. Hette

World Congress on Surface-active Agents, 2,

636-639 (1954) Sodium ethylenediaminetetra-acetate (E.D.T.A., 10%) + polyphosphate (95–90%) gives immediate cold action, can sequester Fe, Cu, and Mn, and supports the failing action of polyphosphates about 90°c. E.D.T.A. salts can decontaminate radioactive dust.

### III-CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Toxic Effects of Synthetic Surfactants

M. Gaultier and E. Fournier
World Congress on Surface-active Agents, 2, 793-809 (1954)

Examination was by oral administration, intravenous, subcutaneous, intramuscular and intraperitoneal injections, patch tests, and mucous-membrane application. In general, non-ionics and anionics show feeble toxicity, but 2-5 g./kg. doses may be dangerous to children. Certain cationics are lethal at 200 mg./kg. Preliminary results of investigations into bronchial poisoning due to their use in laying dust (silicosis prevention) are favourable. The danger in application to the skin or mucous membranes appears small, but attention is drawn to the more real danger of concentrated, alkaline products. Known real danger of concentrated, alkaline produces.

allergens do not appear to be intensified by wetting 8.R.C.

Effects of Detergents on the Skin
E. Sidi, G. R. Melki, and M. Hineky
World Congress on Surface-active Agents, 3, 1309-1317 (1954)

There are collective effects and idiosyncrasies. In the former, the alkalinity of the detergent appears to be the major cause of irritation. In addition there are a "soap effect", resulting in lesions with oedema distinct from eczema, a "shampooing effect", caustic vesicular effect, and a necrotic effect. Individual intolerances are shown by typical eczema and infected lesions. Rubber gloves are recommended, but can cause eczema themselves. Barrier creams are discussed. 8.R.C.

#### Non-ionic Products in the Treatment of Hydrophobic Fibres

Dr. Corbière

World Congress on Surface-active Agents, 2, 487-494 (1954)

A short review covering self-emulsifying antistatic sizes and fibre lubricants, scouring and dyebath assistants, and carriers. For dyeing Tergal an emulsion of 1 part carrier, 0-1 part 40-mol. ethylene oxide-castor oil, and 0-1 part 20-mol. ethylene oxide-oleyl-cetyl condensates is recom-S.R.C.

### Spray-drying of Synthetic Detergents in Theory and Practice

H. Stüpel and A. v. Segesser

World Congress on Surface-active Agents, 1, 402-407 (1954)

Formulae are given for calculation of bead size, and the complications introduced by added builders are discussed.

### Drying Technique for producing Detergents as **Dust-free Hollow Beads**

W. Hauschild

World Congress on Surface-active Agents, 1, 408-413 (1954)

The method of dispersion of the slurry, the drying air, the drying tower, and the collection are discussed. With-SRC or counter-current methods are possible.

### Production of Teepol

P. Dosso

World Congress on Surface-active Agents, 1, 373-378 (1954)

A short description with flow sheet of the Petit-Couronne factory producing 25,000 metric tons of 21% active per S.R.C.

### Use of Fluorescent Brightening Agents in the **Detergent Industry**

A. E. Siegrist

World Congress on Surface-active Agents, 2,

512-521 (1954) The agent should have affinity for the largest number of

substrates. The degree of brightening should be high, but different hues are preferred in different countries. Other considerations are the absence of effect on the colour of the detergent, resistance to alkali, chlorine and polyphosphates, and good solubility and levelling power. behaviour of 16 patented brightening agents on cotton, viscose rayon staple, cellulose acetate, wool, and nylon is tabulated (brightening versus concentration), and also that on cotton in presence of soap, H<sub>2</sub>O<sub>3</sub>, and hypochlorite. S.R.C.

### Synthesis and Reactions of Vinyl Compounds of Silicon. II-Vinylation of Hydrogen-containing Halogenosilanes and Alkyldihalogenosilanes by the Contact Method

M. F. Shostakovskii and D. A. Kochkin

Izvestiya Akad. Nauk S.S.S.R., otdel. khim. nauk, 1150-1152 (Sept. 1956)

PATENTS

Textile Lubricant

Celanese Corpn. of America BP 767,169
A mixture of mineral oil, bodied sperm oil, a partial or high-fatty-alkyl phosphate, an amine and a non-ionic surface-active agent is an excellent lubricant for cellulose acetate and similar fibres. It resists ageing, has antistatic properties and is readily removed during scouring.

Non-ionic Surface-active Agents

Union Carbide & Carbon Corpn. BP 767.851 Manufacture of polyglycol ethers of higher saturated aliphatic monohydric alcohols having powerful wetting properties is described.

Germicidal Detergent Composition

BP 766,401 Compounds of formula Z<sup>1</sup>N·CS·S·S·CS·NZ<sup>2</sup> (Z<sup>1</sup> and Z<sup>2</sup> a single bivalent cycloaliphatic radical or two univalent aliphatic radicals) added to soap and a stabilising agent, e.g. a per compound, give the mixture germicidal properties. C.O.C.

### Polyamino Polycarboxylic Acid Chelating Agents Refined Products Coron. BP 767,162 Refined Products Corpn.

Compounds of formula-

### MOOC-CH<sub>3</sub>·NX·Z·NY-CH<sub>3</sub>·COOM

(X = -CH<sub>2</sub>CH<sub>2</sub>OH, -CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>OH, CH<sub>4</sub>CH(OH)CH<sub>3</sub>, CH<sub>2</sub>CH(OH)CH<sub>4</sub>OH or CH<sub>2</sub>COOM; Y = CH<sub>2</sub>CH<sub>4</sub>OH, CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>OH, CH<sub>2</sub>CH(OH)CH<sub>3</sub> or CH<sub>2</sub>CH(OH)CH<sub>3</sub>OH; Z = CH<sub>4</sub>CH<sub>2</sub> or CH<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>; M = an alkali metal; at least one of X or Y = CH<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>OH, CH<sub>3</sub>CH(OH)CH<sub>3</sub>OH; CH<sub>2</sub>CH(OH)CH<sub>3</sub>OH; CH<sub>2</sub>CH(OH)CH<sub>3</sub>OH; CH<sub>2</sub>CH(OH)CH<sub>3</sub>OH; CH<sub>3</sub>CH(OH)CH<sub>3</sub>OH; CH<sub>3</sub>CH(OH)CH<sub>3</sub>OH or CH4CH(OH)CH4OH), e.g. sodium mono- or diisopropanol ethylenediaminetriacetate, are sequestering agents effective in both alkaline and acid solutions and having antichlor properties.

Aqueous Dispersions

BP 765,885 Th. Goldschmidt More stable dispersions are obtained if the partly divided material to be dispersed is first mixed with alkylene oxide-containing non-ionic surface-active agents or mixtures of such agents with sulphonates, together with water insoluble mono- or poly-carboxylic acids or their water insoluble salts. C.O.C.

**Emulsifying Size** 

H. Eiken

Size is readily emulsified without need for cooking and homogenising it by causing the aqueous slurry to pass through a conical nozzle ending with a parallel-sided slot which directs the liquid on to a plate extending inside the actual body of the sizing liquid, so that the jet impinging on the plate produces in the plate supersonic resonance waves, which are transmitted to the bulk of the liquid. C.O.C.

Flame-retarding Agent for treating Cellulose USP 2,728,680 National Lead Co.

The material is treated with an aqueous solution of tetravalent titanium chloride and a soluble phosphate, the solution gelatinised in the material, treated with an alkaline neutralising agent, rinsed and dried. A better effect is produced if antimony chloride is also present in the impregnating solution. Preferably there should be per I part of titanium 1.5 parts of chlorine and 0.05-0.08 part of phosphorus.

USP 2,728,691

An aqueous solution of tetravalent and trivalent titanium chlorides is similarly used. C.O.C.

### Alkali-metal Borohydrides as Hair-waving Compositions Gillette Co.

Solutions of alkali-metal borohydrides of empirical formula MBH, (M = alkali metal), particularly potassium borohydride, can be used as hairwaving compositions at pH 8.5-11.0 without damaging the hair. C.O.C.

Stabilisation of Rosin Size

Hercules Powder Co. BP 759,513 Addition of the product obtained by reaction of a rosin acid or a rosin derivative containing two ethylenic double bonds in the rosin nucleus with a monoether, monoester, or ether-ester of a gem-diol, or with a gem-diether, or a gem-diester whose ether and/or ester O atoms are attached to acyclic C atoms stabilises paste rosin sizes against crystallisation.

Organophilic Silica Powders

USP 2,728,732 Silica is finely ground and while the surfaces of the particles are still active it is mixed with a polymerisable A product containing 0.05-5.0% ethylenic monomer. carbon is separated from the mixture.

Reducing the Viscosity of Acetone Solutions of Acrylonitrile-Vinylidene Chloride Copolymers

Incorporation of 0.2-2.0% of an inorganic thiocyanate in a solution of an acrylonitrile-vinylidene chloride copolymer reduces its viscosity. The thiocyanate is preferably added to the acetone before the copolymer is C.O.C.

Alkylated Catechol as a Polymerisation Inhibitor

Pittsburgh Plate Glass Co. Alkylated catechol, particularly 3-isopropylcatechol, is useful as a gelation inhibitor in interpolymerisable mixtures of (a) a monomer containing a terminal CH:CH, group and (b) a polyester of a glycol and an  $a\beta$ -ethylenic dicarboxylic acid or a mixture of such an acid and a saturated dicarboxylic acid. It is also useful as an antiskinning agent in paints and varnishes.

Preventing Chemical Degradation of Cellulose during Washing (VII p. 182)

### IV-RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Georg Kalischer

O. Bayer

Chem. Ber., 89, XLIII-LVIII (Dec. 1956)

Activation of Carbon-Carbon Double Bonds by Cationic Catalysts. III—Coloured Species formed by the Interaction of 1:1-Di-p-methoxyphenyl-ethylene with Trichloroacetic Acid in Benzene A. G. Evans, P. M. S. Jones, and J. H. Thomas

J.C.S., 104-111 (Jan. 1957) Spectroscopy shows that two coloured species are formed in the 1:1-di-p-methoxyphenylethylene-trichloroacetic acid-benzene system in the dark, one of which is held to be the classical carbonium ion, by analogy with the spectrum of this olefin in conc. H2SO4, and the other a π-complex between the acid and the olefinic double bond. The results are interpreted in terms of the equilibria-

H+A-

 $3AH + R_2C: CH_2 \rightleftharpoons (CH_3 \cdot CR_2A)_{solv.sAH} \rightleftharpoons R_2C = |CH_2 + 2AH|$ 

where  $R=p\cdot CH_3\cdot O\cdot C_4H_4$  and AH is dimeric trichloroacetic acid. The first equilibrium is set up much more rapidly than the second, while two mol. of acid help in solvating the ion pairs which involve carbonium ions

New and Specific Aromatisation Reaction. Some Further Examples

P. A. Robins and J. Walker

J.C.S., 177-187 (Jan. 1957) The novel aromatisation reaction under the influence of HCl and alcohols of the 1:4-dioxocyclohexane ring which was situated in a cis-syn epd. which fulfilled certain specific structural conditions (cf. J.C.S., 3260 (1956) ) is now shown to be applicable to cis-1:2:3:4:5: 8:9:10-octahydro-1:4-dioxo-6-phenylnaphthalene and its p-methoxy deriv., cis-syn-1:2:3:4:5:6:12:13:14:15-decahydro-1:4-dioxochrysene, and cis-syn-1:2:3:10:1':2':3':4'-octahydro-1':4'-dioxo-1:2-benzofluorene. These epd. fulfil the postulated conditions-(i) that there should be an aromatic nucleus so situated in relation to one of the CO groups of the 1:4-dioxocyclohexane ring as to permit the participation of that CO group in a conjugated system with the aromatic nucleus, so that a halochromic salt may be formed; and (ii) that the CO group not so participating should be converted into a form facilitating the elimination of the O atom in the form of H1O. Further examples of ready ketal formation are described. H.H.H.

Effect of Alkyl Groups on 4-Nitro- and 4-Nitrosophenols W. R. Vaughan and G. K. Finch

J. Org. Chem., 21, 1201-1210 (Nov. 1956)

Some Derivatives of 2-Hydroxy-3-naphthoic Acid H. Pracejus

Annalen, 601, 61-81 (Jan. 1957) Esterification, nitration, treatment with diazomethane, and catalytic hydrogenation lead to 1-amino-2-methoxy-3naphthoic acid. Mercuric acetate readily gives the 4acctoxymercuri deriv., which, after protection of the amino group by phthalation, is treated with methanolic The Ullmann reaction of the resultant iodo cpd. yields the dinaphthyl, which is converted into the optically active 1:1'-dinaphthyl-3:4-3':4'-diquinone-2:2'-dicarboxylic acid. Sodium 2-hydroxy-3-naphthoate with potassium nitrosodisulphonate yields 1.(2'-hydroxy-1'-naphthyl)-naphtho 3:4-quinone 2:3'-dicarboxylic acid, which can also be resolved. The dehydrase action of the optical antipodes is investigated.

4-Dimethylamino-3:5-dimethylazobenzene and Carcinogenic Action

L. Horner and H. Müller

Chem. Ber., 89, 2756-2759 (Dec. 1956) The absorption spectrum of the title cpd. is very similar

to that of azobenzene and unlike that of Butter Yellow. It is also less carcinogenic. H.E.N.

Reactions of Organometallic Compounds Diazonium Salts

D. Y. Curtin and J. A. Ursprung

J. Org. Chem., 21, 1221-1225 (Nov. 1956) Alkyl- and aryl-zinc chlorides react with aryldiazonium fluoroborates suspended in ether to give unsymmetrical H.E.N.

Monoazo Derivatives of N-Thiazolyl- and N-Benzothiazolyl-α-furoylacetamides A. Cerniani and R. Passerini

Ricerca sci., 25, 2593-2598 (1955):
Chem. Abs., 50, 17455 (25 Nov. 1956)
Ethyl 2-furoylacetate was boiled with 2-aminothiazole or

2-aminobenzothiazole to give amides, e.g. a-2-furoyl-Nbenzothiazolylacetamide-



These amides dissolve in dil. aq. NaOH, and couple with diazonium salts to give  $\alpha$ -arylazo derivatives. E.S.

Methyl and Ethyl Homologues of 1:7-Dihydroxynaphthalene

Ng. Ph. Buu-Hoi and D. Lavit

J. Org. Chem., 21, 1257-1259 (Nov. 1956) Formylation with dimethylformamide of the dimethyl ether yields mainly the 4-, but also some of the 8-deriv., while Friedel-Crafts acetylation gives the 4-cpd. only. The products are converted into the corresponding alkyl-1:7-dihydroxynaphthalenes. The 4-methyl deriv. transformed also into the 4:8-dimethyl cpd. H.E.N.

### Photoreduction of Triphenylmethane Dyes in the **Bound State**

G. Oster and J. S. Bellin

J. Amer. Chem. Soc., 79, 294-298 (20 Jan. 1957) Triphenylmethane dyes are not photoreduced unless they are bound to water-soluble high polymers. In contrast to their behaviour in the light, reduction of these dyes in the dark by strong reducing agents is actually inhibited when they are bound. The photochemical behaviour of the bound dyes differs in several respects from that of free fluorescein-type dyes. With the latter, when free, the quantum yield of photoreduction decrease with increase in dye concentration, whereas with bound dyes the quantum yield increases with increase in dye concentration. Nitrobenzene retards the rate of fading of free dyes, while in the case of bound dyes fading is inhibited until all the nitrobenzene is used up and then fading proceeds at the same rate as in an inhibitor-free Analysis of the kinetic data shows that bound long-lived excited dye molecules react with the reductant to give colourless products. Interaction between bound dye molecules in the ground state and those in the first electronically excited singlet state is the principal mechanism for formation of long-lived excited dye molecules. This is confirmed by the fact that self-quenching of the fluorescence of bound dye molecules occurs at abnormally low concentrations. An analogy is drawn between these systems and those occurring in photosynthetic systems. CO.C.

### Polymethin Dyes from Heterocyclic Derivatives of Pyruvic Acid-II

W. Franke

Chem. Ber., 89, 2727-2734 (Dec. 1956)

Oximes of heterocyclic pyruvic acids condense with certain heterocyclic aldehydes or their anils in anhydride. The aldehydes are of the type R:CH-CHO the products of the general formula R'-C(CN):CH-CH:R, R and R' being heterocyclic nuclei.

### Solvent Quenching of Fluorescence

E. J. Bowen and D. M. Stebbens J.C.S., 360–363 (Jan. 1957)

### o-Quinones. IX-Preparation and Properties of

some Amino Derivatives L. Horner and H. Lang

### Chem. Ber., 89, 2769-2773 (Dec. 1956) Some Hydroxyfluorene Derivatives

H. Bryant and E. Sawicki

J. Org. Chem., 21, 1322-1324 (Nov. 1956)

### Some Reactions of 1-Anthrol

S. Lele, N. H. Shah, and S. Sethna J. Org. Chem., 21, 1293-1295 (Nov. 1956)

### trans-Fluorenacenedione, a New, Vattable Diketone F. Ebel and W. Deuschel

Chem. Ber., 89, 2794-2799 (Dec. 1956)

The title cpd. (I) is obtained in 65% yield from p-xylene by reaction with cyclohexene and AlCl<sub>3</sub>, followed by

dehydrogenation, oxidation to the dicarboxylic acid, and ring-closure. It is red-violet, giving a blue vat. The following deriv. are described: the red dinitro, green diamino, red dibromo, and the violet-red 2:7-dicarboxylic acid, its red acid dichloride, and pale red dianilide. deriv. yield blue vats.

### 10:11-Diaza-trans-fluorenacenedione

Ibid., 2799-2807 This cpd. is obtained as brown-violet needles in 60% yield from indanone by nitrosation with amyl nitrite, followed by oxidation with dichromate-acetic acid, but can be synthesised also in 12-14% yield from propio-phenone. It yields a blue vat. Various deriv. are phenone. It yields a blue vat. Various deriv. are prepared. The red-brown bisbenzoylamino epd. gives a blue, difficultly sol. vat salt; the brown monobenzoylamino deriv, a blue vat, which dyes cotton weakly yellowbrown.

### Polybenzofluoranthenes

K. F. Lang and E.-A. Theiling Chem. Ber., 89, 2734–2737 (Dec. 1956)

Aromatic Hydrocarbons LXXVIII—Higher Condensed Pyrenes

E. Clar and O. Kühn

Annalen, 601, 181-192 (Jan. 1957)

Naphtho-1':7'-2:4-perylene is isolated from the products of the interaction of 1-bromonaphthalene and pyrene in NaCl-AlCl<sub>3</sub>. Condensation with maleic anhydride followed by decarboxylation yields pyreno-1':9'-4:6-pyrene (1). 3:3'-Dipyrenyl, synthesised from 3-bromo-pyrene and copper powder, is cyclised by means of a 4:4'-bond. Condensation with maleic anhydride followed by decarboxylation yields naphtho-2':8'-3:5-pyreno-3": By condensation of pyrene with 5"-10:8-pyrene (II). 2 mol. of 1-bromonaphthalene followed by reaction with 2 mol. of maleic anhydride and decarboxylation, dipyreno-3':5'-5:3-3'':5''-10:8-pyrene (III) is obtained. The spectra of the series naphthalene, pyrene, 1:12-benzoperylene, I, II, and III are compared.

### LXXIX-1:2-5:6-Dibenzoperylene and 2:3-6:7-

10:11-Tribenzor uoranthene E. Clar and W. Willieks

Ibid., 193-201 H.E.N.

### Chlorination of Copper Phthalocyanine V. F. Borodkin and K. V. Usacheva

J. Appl. Chem. U.S.S.R., 29, 1383-1387

(Sept. 1956)

During an investigation of conditions of chlorination of Cu phthalocyanine in solution and in the molten state it was found that a highly chlorinated compound is best prepared in molten AlCl<sub>2</sub> or NaCl. The presence of moisture in the reaction mass leads to destruction of the phthalocyanine. T.Z.W.

### Anthochlor Pigments of Coreopsis tinctoria

M. Shimikoriyama

J. Amer. Chem. Soc., 79, 214-220 (5 Jan. 1957)

Two anthochlor pigments have been isolated from the ray flowers of Coreopsis tinctoria. These two orangeyellow pigments have been named marcin and maritimein, and their constitutions proved to be 4'-glucosidoxy-2':3':3:4-tetrahydroxychalcone and 6-glucosidoxy-7:3':4'-trihydroxyaurone respectively.

### Effect of Roasting Schedule on the Colour of Red Ferric Oxide Pigment prepared from Copperas L. N. Uspenskaya and A. Kh. Girenko

J. Appl. Chem. U.S.S.R., 29, 1040-1044 (July 1956) For the preparation of a ferric oxide pigment that is as close to red in hue as possible, the copperas should be dried at 80-100°c. and then heated so that the temp. rises at 2.5-5 c. per min. to a final temp. of 750-800 c. Lower and higher final temp, are detrimental to the colour. The fineness of the copperas is of no importance; neither is the rate of cooling.

### Chemistry of the Coloured Glasses prepared by

M. V. Lomonosov Yu. V. Morachevskii and R. A. Shtrikhman

J. Appl. Chem. U.S.S.R., 29, 1432-1435

(Sept. 1956)

A study of the scientific legacy of M. V. Lomonosov, dealing with the preparation of coloured glasses, is made, giving the analytical data for their colouring components. T.Z.W.

### PATENTS

### Salicylic Ester and Amide Colour Couplers

Eastman Kodak Co. USP 2,728,660

Salicylic amides, anilides, and esters having an electron-donating substituent in the nuclear position para to the amide or ester group have greatly improved coupling rates in colour development processes and yield much more stable dye images than do either salicylic acid or unsubstituted salicylic amides. Č.O.C.

### Benzoylacetanilide Colour Couplers

Eastman Kodak Co.

Compounds of formula-

USP 2,728,658



(R = subst, or unsubst, mononuclear Ar), e.g.—

are better colour couplers than the o-alkoxybenzoyl-acetanilides described in USP 2,407,210, as they have better coupling ability and do not decrease the tinetonial power of the dyo.

C.O.C.

### Resin Colour Couplers

Kodak Resin colour couplers of formula—

$$BP\ 767,028$$

$$\begin{bmatrix} -\mathrm{CH}_{2}\cdot \{\mathrm{CHR}^{1}\}_{z}\cdot \mathrm{CR}^{2}(\mathrm{COOH})\cdot [\mathrm{CH}_{z}]_{y} & \mathrm{CO} \\ -\mathrm{CH}_{2}\cdot \{\mathrm{CHR}^{1}\}_{z}\cdot \mathrm{CR}^{2}(\mathrm{COOH})\cdot [\mathrm{CH}_{z}]_{y} & \mathrm{CO} \cdot \mathrm{NHR}^{2} \end{bmatrix}_{y}$$

(R<sup>1</sup> = H, phenyl, Alk, alkoxy, carboxyalkyl or acyloxy; x = 0 when y = 1 or x = 1 when y = 0; R<sup>2</sup> = an organic radical having a coupling function; R<sup>3</sup> = H or Alk) may be directly incorporated in colour-sensitised emulsions.

### Metal(Chromium and Cobalt)-complex Monoazo Dyes for Wool, Nylon, etc.

BP 765,355

1:2 Metal complexes of oo'-dihydroxy-, σ-earboxy-σ'-hydroxy-, σ-σ-ammo-σ'-hydroxy-monoszo dyes are made by combining suitable 1:1 metal complexes with metal-free dyes, the compounds used containing no sulphuric ester, sulphamyl, or sulphone groups, and one of them (preferably the 1:1 metal complex) containing one SO<sub>3</sub>H group. The metals employed are Cr and Co, and the products give blacks, greys, browns, greens, and navy blues on wool, nylon, etc., from neutral or weakly acid baths. Thus the 1:1 Cr complex of the monoszo compound 1-amino-2-naphthol-4-sulphonic acid-β-naphthol is heated in aq. Na<sub>2</sub>CO<sub>3</sub> with 1 mol. of the monoszo compound 2-amino-5-nitrophenol-β-naphthol at 90-95°c, for 1 hr. The product dyes wool navy blue.

### Metal(Chromium)-complex Monoazo Dyes for Wool, Nylon, etc.

Ciba

Complexes from 1 atom of Cr and 2 mol. of oo'-dihydroxy monoazo dyes free from COOH and SO<sub>3</sub>H groups, at least one of which has the structure—

(R = an ortho-coupling keto-methylene residue;  $Z^1$  and  $Z^2$  = H, Alk or OAlk of < 7 C) dye wool, nylon, etc., from neutral, weakly alkaline, or weakly acid baths. Thus 2-aminophenol-4-sulphon-m-sulphamylanilide is diazotised and coupled with 3-methyl-1-phenyl-5-pyrazolone, and the product boiled with sodium chromosalicylate. The 1:2 chromium complex so formed dyes wool orange from a weakly acid or neutral bath. E.S.

### Metal(Chromium)-complex Monoazo and Azamethin Dyes for Wool, Nylon, etc. BP 766,402

The sparingly soluble 1:1 chromium complexes of monoazo and azamethin dyes free from SO<sub>3</sub>H and COOH groups, and containing at least one SO<sub>3</sub>Alk group, react with soln. of organic compounds capable of metal-complex formation to give more soluble complexes. The products level better than the similar dyes of BP 692,073 (cf. J.S.D.C., 69, 306 (1953)) which do not contain SO<sub>3</sub>Alk groups. Thus the 1:1 chromium complex of the monoazo dye 4-methylsulphonylanthranilic acid—3-methyl-1-phenyl-5-pyrazolone is boiled in methanol with the 5-sulphonanilide of salicylic acid in presence of sodium acetate. The product dyes wool level, pure yellow from a weakly acid bath. E.S.

### Blue Metal(Copper)-complex Disazo Direct Dyes Ciba BP 766,014

The copper complexes of disazo compounds -

(X = OH, OAlk, or Hal; R¹ and R² = Alk, hydroxyalkyl, or alkenyl) are very greenish-blue direct dyes. Thus tetrazotised dianisidine is coupled with 2 mol, of N- $\beta$ -hydroxyethyl-H acid in presence of Na $_2$ CO $_2$ , and the product is coppered, with demethylation of the OCH $_2$  groups of the dianisidine residue, by refluxing with aq. ammoniacal CuSO $_1$  in presence of monoethanolamine.

### Metal(Cobalt and Chromium)-complex Disazo Dyes for Wool, Nylon, etc.

y BP 766,018 The cobalt and chromium complexes of disazo dyes—

(A¹, A² = ortho-coupling phenols, naphthols or enolic compounds; R¹, R², R³, R³ = H, Alk, or constituents of organic carboxylic ring systems) dye wool from a neutral or weakly acid bath if SO<sub>3</sub>H groups are excluded, but require a more strongly acid bath if SO<sub>3</sub>H groups are present. Thus ethylenediamine is diacylated with ontrobenzenesulphonyl chloride, and the nitro groups are then reduced to amino groups. The product is tetrazotised and coupled with 2 mol. of  $\beta$ -naphthol, to give

which is dissolved in ethylene glycol, made alkaline with aq. NaOH, and heated at 120–130°C, with chromic acetate. The chromium complex so formed dyes wool red-brown from a weakly acid bath. E.S.

### Yellow Disazo Stilbene Direct Dyes

FBy
BP 766,020
Brilliant Yellow (C.I. Direct Yellow 4) (I) is alkylated
using dihalogeno ethers or acetals to give direct dyes
brighter than Chrysophenine and of better wet fastness.
Thus I is dissolved in aq. alcoholic NaOH, Na<sub>4</sub>CO<sub>5</sub> and
2:2'-dichlorodicthyl ether are added, and the mixture is
heated in an autoclave at 90–97 c, for 48–60 hr., to give—

### Bright Red Disazo Direct Dyes having a Triazine Ring BP 766,015

Disazo compounds

(R = benzene-series radical containing a water-solubilising group, e.g.  $SO_3H$ , COOH,  $SO_2NH_2$ ,  $SO_2Alk$ ; X = Hal, OH,  $NH_2$  or residue of primary or see, amine) dye cellulose bright scarlet and red. Thus diazotised 4-aminoazobenzene-3.4'-disulphonic acid is coupled with N-acetyl-J

acid under weakly acid conditions, the by-product coupled in the 8-position is separated, and the acetyl group is removed by hydrolysis. The aminodisazo dye so formed is condensed at  $0-3^{\circ}\mathrm{C}$ , with 1 mol. of cyanuric chloride in presence of  $\mathrm{Na_{2}Co_{3}}$ . The second Cl of the triazine ring is then condensed with metanilamide at  $45^{\circ}\mathrm{C}$ ., and the third Cl with aniline at  $95^{\circ}\mathrm{C}$ . to give the desired dye (R =  $m\cdot\mathrm{C_{6}H_{6}}\cdot\mathrm{SO_{2}}\cdot\mathrm{NH_{2}}$ ; X =  $\mathrm{NH\cdot\mathrm{C_{6}H_{5}}}$ ), which dyes cotton vivid red. E.S.

### Metallisable Trisazo Stilbene Direct Dyes

Aminomonoazo compounds-

$$z$$
CH:CH
N:N
 $\begin{pmatrix} s \\ h \end{pmatrix}$ 
HO<sub>2</sub>S

 $(x = \text{NO}_9, \text{ NH-Acyl}, \text{ or a naphthylenetriazole group in which the naphthalene residue is connected in the 1:2-position with the triazole ring; <math>y = \text{COOH}, \text{ OCH}_2$  or  $\text{OC}_2\text{H}_5$ ;  $w = \text{H}, \text{ lower Alk}, \text{ lower OAlk}, \text{ or NH-Acyl}; \text{ the azo group is in position 4 or 5) are diazotised and coupled with monoazo compounds—$ 

(one  $z \approx \mathrm{OH}$ , the other = N:N·R; R = residue of benzene or naphthalene series having OH or COOH ortho to the azo group) or a metal complex thereof, coupling taking place at position 6. The trisazo compounds so formed are converted into Cu or Ni complexes in substance or on the fibre. Thus the monoazo compound anthranilic acid  $\rightarrow$  J acid is treated according to the method of BP 697.488 (J.S.D.C., 69, 463 (1953)), replacing the NH<sub>2</sub> group by OH, and then made into the Cu complex, which is coupled with the diazo compound of the aminomonoazo compound 4 · amino · 4' · nitrostilbene · 2 : 2' · disulphonic acid  $\rightarrow$  p·oresidine, to give—

Treatment at 90°C. with aq. ammoniacal CuO in presence of diethanolamine yields the Cu complex, which dyes cellulose greenish blue.

### 1-Hydroxy-4-(2'-cyano)-ethylaminoanthraquinone— Disperse Dye fast to Gas Fumes

Disperse Dye fast to Gas Fumes

8

BP 765,725

This red-violet disperse dye fast to gas fumes and light

This red-violet disperse dye fast to gas fumes and light is obtained by condensing 2-cyanoethylamine with 1:4-dihydroxyanthraquinone in a low-boiling alcohol solvent under reflux.

E.T.

### Anthraquinone Disperse Dye Mixtures

Disperse dyes of the 1-hydroxy-4-phenylaminoanthraquinone series which have little affinity for acetate rayon show greatly enhanced affinity in the form of mixtures. These mixtures contain not more than 2 parts by weight of 1-hydroxy-4-phenylaminoanthraquinone and not less than 1 part of a dye of formula—

 $(R^1=H \text{ or } CH_a;\ R^1=COO\cdot CH_a,\ COO\cdot C_aH_a,\ CO\cdot H \text{ or } CO\cdot CH_aY;\ Y=H,\ CI \text{ or alkoxy}).$  E.T.

### Dyes for Polyvinyl-derivative Fibres

Fran \$BP 765,356 Anthraquinone dyes containing one or more groups of formula  $NH\cdot[Alkylene\cdot O]_n\cdot CO\cdot R$  (n = a whole number;

B = H or Alk of < 4 C) have good affinity for fibres formed from polyvinyl derivatives. Thus Rhovyl is dyed blue with acetoxyethylaminoanthraquinone applied at 40–42°C, in an aqueous bath in presence of a mixture of p-chlorotoluene and a lauryl alcohol-ethylene oxide condensate.

### Substituted Acylaminoanthraquinone Vat Dye

Fast red and bluish-red dves of formula-

(X = sulphonic-acid dialkylamide or dialkyleneamide whose alkylene groups form a ring; benzene group I may contain substituents other than the aforementioned groups; Y = same or different Hal) are produced by condensing 1 benzoylamino 4 amino 6:7 dihalogeno anthraquinone with a benzoylating agent containing such groups as

$$\cdot \mathrm{SO_3N} \overset{\mathrm{CH_4CH_4}}{\underset{\mathrm{CH_4CH_4}}{\mathrm{CH_3}}} \quad \mathrm{or} \quad \cdot \mathrm{SO_3N} \overset{\mathrm{CH_4CH_4}}{\underset{\mathrm{CH_4CH_4}}{\mathrm{CH_4CH_4}}}$$

in the m- or p- position to the 'NHCO· group. These dyes give bluer dyeings and stronger prints on cotton and viscose rayon than the similar dyes obtained by the process of BP 605,466 (J.S.D.C., 65, 34 (1949)). E.T.

### Anthraquinone Acid Dye

FBy
4-Berzylcyclohexylamine is condensed with quinizarin or leucoquinizarin by heating in presence or absence of a solvent. The leuco-compound is oxidised in boiling nitrobenzene in presence of a small amount of piperidine. The separated product is then sulphonated with monohydrate at room temperature to give—

The yield is greater than that obtained by the method of BP 564,859 (J.s.D.C., 61, 80 (1945)), in which the benzyl group is attached to the 2-position of the cyclohexylamine instead of the 4-position. E.T.

### Quaternary Salts of Indigos

unternary Saits of Indigos

eneral Aniline

USP 2,728,774

Ludica and its total belogano and tetra albul deriv, are

Indigo and its tetrahalogeno and tetra-alkyl deriv. are first halogenomethylated, particularly chloromethylated, and then treated with quaternising agents to yield watersoluble blue to green dyes which yield dyeings and prints having excellent fastness to chlorine, washing and light. Thus indigo is slowly stirred into bischloromethyl ether in conc. H<sub>2</sub>SO<sub>4</sub> at 0°c., the solution stirred for 2 hr. and poured over ice. After a short stirring a blue monochloromethylindigo separates out and is filtered off, washed neutral and dried. It is heated on a steam bath for 1 hr. with water and trimethylthiourea. The isothiouronium salt, which quickly forms a thick paste, is diluted with acetone, filtered, the filtrate triturated several times with acetone, refiltered and dried. The water-soluble product yields greener prints on cotton than does indigo. It has

$$CI \left\{ \begin{array}{c} (CH_3)_2 N \\ CCH_4 \\ H_3 CNH \end{array} \right. \left. \begin{array}{c} O & O \\ N \\ H \end{array} \right. \left. \begin{array}{c} O & O \\ N \\ H \end{array} \right. \left. \begin{array}{c} O & O \\ CO & C \end{array} \right.$$

### Dyes of the Tetra-azaporphin Series

BP 762,778

Metal or metal-free tetra-azaporphin derivatives are treated with chloro- or bromo-methylating agents to

introduce -CH, Hal group(s) (cf. BP 689,153-J.S.D.C., 69, 216 (1953)). The resulting compounds are then treated with phenols so that the final product contains one or more CH<sub>2</sub>ArOH groups. Thus, nickel tetra-(chloromethyl)-tetramethyldibenzotetra-azaporphin, anhydrous ZnCl<sub>2</sub> and methyl salicylate are heated at 165°c, for 3 hr. The excess methyl salicylate is removed by steam distillation. The blue product obtained by filtration is heated in  $\beta$ -ethoxyethanol and KOH at 120°c. for 90 min. to hydrolyse the ester group. The final product is precipitated by HCl, washed free from acid and dried. It is used as a lake-forming or chromable dye.

Pigments of the Tetra-azaporphin Series BP 762,783

Blue pigments are produced by heating a substituted 2-amino-5-iminopyrroline with an unsaturated dinitrile in a diluent, e.g. pyridine, dioxan, ethanol, etc. One of the tautomeric forms of the 2-amino-5-iminopyrroline is

(R1 = H, subst. or unsubst. Alk, Ar, aralkyl or cycloalkyl radical; R<sup>2</sup> = subst. or unsubst. Alk, Ar, aralkyl or cycloalkyl radical; R<sup>1</sup> and R<sup>2</sup> may be joined to form a cycloalkyl or cycloalkylene ring with the 3- and 4-C atoms of the pyrroline ring). Thus, hexahydro-1-amino-1-imino-isoindolenine and phthalonitrile are heated for 30 hr. in boiling anhydrous methanol. The blue pigment resulting is essentially monocyclohexenotribenzotetra-E.T.

Phthalocyanine-Tetra-azaporphin Mixtures-Vat Dyes

ICI BP 763,084

Metal phthalocyanines, e.g. Co, Fe, Sn, V, which are not readily vatted with alkaline hydrosulphite solutions are milled with tetra-azaporphin derivatives containing au acid or acid halide group in presence of a dispersing agent to give readily-vattable dye pastes. The component of the mixture which is free from acid groups comprises 50-95% of the mixture. Thus, an aqueous milled paste containing cobalt phthalocyanine (5 parts) and a small amount of dispersing agent is mixed with a similar paste containing the sodium salt of a partially sulphonated cobalt dicyclohexenodibenzotetra-aza-porphin (approx. 1-66). The mixed dye paste gives an olive-green vat which dyes cotton a clear blue. E T

### Phthalocyanine Lakes

General Aniline Pigments of very good texture and rheological properties are obtained by forming the water-insoluble alkaline-earth metal lakes of water-insoluble phthalocyanine dyes in presence of < 0.01 part of a 1-hydroxyethylglyoxalidine containing a  $C_{s-1s}$  hydrocarbon chain in the 2-position or  $\prec$  0-1 part of an ammonium salt of an acid not weaker than acetic acid or a mixture of the two. Thus copper hexadecachlorophthalocyanine acid-pasted press-cake is made into an aqueous slurry. This is brought to pH made into an aqueous sturry. This is brought to pH 7.5–8.0 with ammonia, ammonium chloride added and the mixture heated to 95°C. An aqueous solution of wood rosin and NaOH is added to the hot slurry, which is stirred at 95–98°C. for 30 min. and then aq. BaCl<sub>2</sub> added. The slurry is boiled for 30 min. and cooled to 65°C. by adding water. The product is filtered off, washed and dried. It is much superior in texture to the product obtained without addition of anymonium chloride.

### Heterocyclyl Ketone Dyes

without addition of ammonium chloride.

Kodak Dyes of formula-

C.O.C. BP 767,262

(R<sup>1</sup> = Alk; R<sup>2</sup> = Alk or allyl; Z = atoms to complete a 5- or 6-membered ring; n=0 or 1), e.g. 3-carboxymethyl-5-(3-ethyl-2-benzothiazolyidene)acetyl-4-hydroxy-2thionothiazoline, are obtained by treating dyes of formula

( $R^3$  = alkoxy), made e.g. by the process described in FP 1,018,590, with aqueous NaOH. C.O.C.

Cyanine Dyes Hord

RP 766.380

Dyes of formula—

 $(R^{1} - Alk; R^{2} - Alk, Ar \text{ or thioether}; R^{3} - \text{phenyl or naphthyl substituted by Alk, alkoxy or Hal; } R^{4} - Alk; X - acid radical; D^{1} - atoms to complete a hetero$ cyclic ring; D<sup>2</sup> = atoms to complete a heterocyclic N cycuc ring; D\* = atoms to complete a heterocyclic N ketomethylene ring; m and n = 0 or 1), e.g. [1-methyl-3-methylthio-4-phenyl-1:2:4-triazole-5-][3'-ethylbenzo-thiazole-2'-]trimethineyanine iodide, are obtained by treating a compound of formula—

$$\begin{array}{c|c} R^{I \cdot C} & N - NR^{I} X \\ N - C \cdot CH_{I} \\ R^{I} \end{array}$$

with one of formula-

(Q = thioether or thioethervinyl) or

 $(R^6 = Alk).$ COC

Trinuclear meroCyanine Dyes containing a Chain Alkoxy Group

Eastman Kodak Co. USP 2,728,766 Dves of formula-

 $(R^1 \text{ and } R^4 = Alk; R^2 = H \text{ or } Alk; R^3 = H, Alk, Ar \text{ or }$ alkoxy; J1 and J2 together = atoms to complete a thiazolidone nucleus; Z and Q = atoms to complete a 5- or 6-membered nucleus), e.g. 2-[2-(3-carboxymethyl-4oxo-2-thiono-5-thiazolidylidene-2-ethoxyethylidene (3-ethyl-2(3)-benzothiazolylidene)ethylidene 3-methyl-5-thiazolidone, have photosensitising properties. examples of their preparation are given.

Zinc and Cadmium Sulphides and Selenides

BP 768,079 High yields of excellent purity are obtained by passing gaseous hydrogen sulphide or scienide into a solution of a zine or cadmium salt of an organic acid or a zine or cadmium complex ammonium salt in an alkanol of 1-5 C while maintaining the solution under reflux.

Improving the Surface Properties of Pigments Dehydag Deutsche Hydrierwerke

Pigments are made more readily wettable by and dispersible in liquids by impregnating them with a mix-ture of a metal salt of a sulphonic acid or of an acid sulphuric ester and a salt of ammonia or an organic base with such acids or esters.

Triarylmethane Compounds as Redox Indicators in the Schoenemann Reaction, I.—Mechanism of the Schoenemann Reaction (XIV p. 187)

### V-PAINTS; ENAMELS; INKS

Marking Ink

BP 764,863

R. P. Frage: A marking ink resistant to laundering and bleaching consists of carbon black and/or one or more chemically inert water-insoluble metallic compounds, e.g. rouge, and sulphur dispersed in a liquid of vapour pressure < 0.11 mm. Hg at 20 c., e.g. mineral oil or a glycol.

COC

Photogravure Inks

Sun Printers

BP 768 228

Ink of excellent flow qualities is obtained by adding a pigment or dye to a synthetic resin dissolved or dispersed in a volatile solvent so that the ink has viscosity 0-04-7.0 poises and then agitating it with water to form a water-in-oil emulsion. Such an ink is readily transferred to the uneven surface of inferior paper, gives smoothness in both lightly and heavily etched parts of a print, and can produce matt or glossy prints which are not affected by moisture.

**Emulsion Paints** 

Distillers Co. BP 767,729

Stable emulsions yielding coatings from which the plasticiser does not tend to migrate comprise an aqueous dispersion of a copolymer of styrene or vinyl acetate with an alkyl diester of an αβ-unsaturated dicarboxylic acid, an organic hydrophilic colloid as stabiliser, and a pigment.

Thickening Aqueous Dispersions of Polymers of Alkoxyalkyl Methacrylates

BP 764,021 Rohm & Haas

Aqueous dispersions of polymers containing an acidic component, e.g. acrylic acid, and a preponderating amount of a compound of formula  $H_2\text{CC}(\text{CH}_2)\text{CO-O}(\text{CH}_2)\text{CK}$  (x=a whole number; R=Alk, Ar, aralkyl or alkoxy and may be attached to the neighbouring  $\text{CH}_2$  group or groups to form a ring), e.g. 2-methoxyethyl methacrylate or tetrahydrofurfuryl methacrylate, are thickened by addition of basic substances, e.g. NH<sub>4</sub>OH. Such thickened dispersions can be used as leather finishes, stove lacquers,

Improving the Corrosion Resistance of Stoving Enamels

Newton, Chambers & Co. BP 767,564

The corrosion resistance of a stoving enamel is improved by applying four coats to the metal surface. The first coat is of filled enamel and is lightly baked on to the surface. The second coat is of unfilled enamel and is allowed to dry before the third coat, of filled enamel, is applied and lightly baked. Finally a coat of unfilled enamel is applied and the whole stoved on to the surface. The filler used is a pigment or mixture of pigments, e.g. silica,  $BaSO_4$ ,  $TiO_2$ ,  $Cr_2O_3$ , etc. C.O.C.

Emulsification of Offset Inks (IX p. 183)

### VI-FIBRES: YARNS: FABRICS

Water Transport Mechanisms in Textile Materials. I-Rôle of Yarn Roughness in Capillary-type Penetration

N. R. S. Hollies, M. K. Kaessinger, and H. Bogaty

Text. Remarch J., 26, 829-835 (Nov. 1956) Rates of liquid water transport in yarns (cotton, Dacron, nylon, wool, and wool blends) are directly related to the apparent advancing contact angle of water on the yarns, which in turn depends on the constructional features of the yarns, and only indirectly to the surface properties of single fibres. Increase of yarn roughness due to more random arrangement of the fibres leads to a reduced rate of water transport owing to an increase in contact angle and a decrease in mean length of capillaries.

Crystallite Regularity in Cellulose Fibres

W. O. Statton

J. Polymer Sci., 22, 385-397 (Dec. 1956) Small-angle X-ray scattering studies of cellulose fibres show long spacing order in the direction of the fibre axis. It is suggested that the crystallites act as diffracting centres to form a linear lattice. It is also suggested that diffuse

scattering at small angles is largely due to micro-voids which are elongated in the direction of the fibre axis.

Electron-microscopic Investigation of the Formation of Fibrillar Structures from Viscose

O. Samuelson, F. Alvang, and A. Svensson Svensk Papperstidning, 59, 712-714 (31 Oct. 1956) A dilute solution of cellulose acetate in acetone was shown to be molecularly dispersed by means of osmotic measurements. The cellulose acetate was then saponified, and the cellulose powder obtained was converted into viscose. Model filaments made from this viscose exhibited the same properties as normal viscose rayon filaments. After ultrasonic irradiation a fibrillar structure could be observed with an electron microscope.

Fault in Viscose Rayon-Cellulose Acetate Fabric due to Action of Light during Storage

N. Iwanow and R. Schneider

Bull. Inst. Text. France, (63), 7-13 (Oct. 1956) A fabric made from a blend of viscose rayon and TiO<sub>3</sub>delustred cellulose acetate developed a light streak along the folds exposed to light during a storage period of several months. The effect arose from an apparent increase in pigmentation of the cellulose acetate fibres due to the formation of granules of oxidised cellulose, possessing a refractive index different from that of normal cellulose, at the boundaries of TiO2 particles. The authors' method of examining fabric samples immersed in liquid paraffin (cf. J.s.D.c., 72, 133 (1956)) proved of value in tracing the cause of this fault. J.C.F.

Separation of Ortho- and Para-cortical Cells from Wool

M. Leveau

Bull. Inst. Text. France, (63), 91-96 (Oct. 1956) Merino wool, treated with 6 N-HCl for 1 hr. at 60°C. and rinsed, is macerated with water for I min., and the suspension filtered through wire gauze (100 meshes/cm.2), The material passing through the filter is macerated for a further 15 min, and passed through a No. 2 sintered-glass filter, which retains orthocortical cells containing < 6% of the para component, and allows cuticular material to pass through. To obtain paracortical cells, the fibrous residue retained by the gauze in the first filtration is macerated for 9 min., and again filtered through gauze. The residue on the filter is treated with cold 1% for 1 min., rinsed, suspended in water, and filtered through gauze. Paracortical material in fibrous form is retained by the gauze.

Progress in Man-made Protein Fibres

J. S. Gillespie

Text. Research J., 26, 881-888 (Nov. 1956) A review covering protein raw materials, fibre manufacture, with particular reference to zein fibre, dyeing properties, and applications.

Physicochemical Properties of Synthetic Fibres

A. A. Konkin and G. I. Kudryavtsev Tekstil. prom., 16, 9-14 (Aug. 1956) Physicochemical properties of synthetic fibres in current production in the U.S.S.R. and elsewhere are given in the form of tables and graphs, and the comparative merits of

the fibres are discussed from this point of view. A.E.S.

Density, Infrared Absorption, and Crystallinity of 6,6- and 6,10-Nylons H. W. S. Starkweather and R. E. Moynihan

J. Polymer Sci., 22, 363-368 (Dec. 1956) Intensities of crystalline and amorphous infrared bands of nylon films are plotted against density, and the curves extrapolated to zero intensity to give values for the amorphous and crystalline densities. The amorphous amorphous and crystalline densities. The amorphous density of 6,10-nylon is in agreement with a previous value for quenched films, but that of 6,6-nylon is about 2% lower. Crystalline densities are somewhat lower than W.R.M. those obtained from X-ray data.

Effect of Drawing on Infrared Dichroism of 6,6-Nylon

G. Caroti and J. H. Dusenbury

J. Polymer Sci., 22, 399-407 (Dec. 1956) Dichroic ratios obtained from infrared absorption measurements in the 3-µ, region on 6,6-nylon suggest that there is a random arrangement of chains at a draw ratio of unity and that the chains become increasingly aligned in the direction of the filament axis as drawing increases. Results from X-ray diffraction studies are consistent with these views.

### Action of Hydrazine on Terylene

J. C. Atkinson and J. B. Speakman

Chem, and Ind., 74 (19 Jan. 1957) Terylene is unusually sensitive to attack by hydrazine. At 100°c, complete destruction is brought about by a 2% solution in butanol in 60 min. and by a 10% soln, in 5 min. The ester linkages of Terylene undergo hydrazinolysis much more easily than the peptide links of keratin. Wool may be isolated and recovered by treating rags containing Terylene with a solution of hydrazine in butanol (or other solvent). W.R.M.

### Polyacrylonitrile Fibre (Nitron)

Tekstil. prom., 16, 16-19 (Nov. 1956)

Details are given of a method of preparing polyacrylonitrile for use in fibre manufacture. The monomer is

polymerised in unstirred aq. soln. in presence of potassium persulphate, sodium hydrosulphite, and sulphuric acid.

### Acrylic Fibres. XVII-Binary and Ternary Copolymers of Acrylonitrile H. Reichert and A. Hunyar

Faserforsch, und Textiltech., 7, 546-556 (Dec. 1956) Fibres spun from copolymers of acrylonitrile and a vinylpyridine or methacrylic acid show essentially the same physical and textile properties as those from polyacrylonitrile but give deeper colours with acid or basic dyes. Copolymerisation with methyl methacrylate as a third component improves fibre properties. W.R.M. third component improves fibre properties.

### Photodecomposition of Polyvinyl Chloride Fibre-I T. Imoto and Y. Ogo

J. Soc. Textile Cellulose Ind. Japan, 12,

823-827 (Nov. 1956)

The rate of decomposition of polyvinyl chloride fibre by ultraviolet radiation in vacuo is

$$v_p = KP_a \neq (I)$$

 $(K={
m constant};\ P_a={
m amorphous}\ {
m region}\ {
m content}\ {
m in}\ {
m sample}\ {
m fibre};\ {
m and}\ I={
m intensity}\ {
m of}\ {
m absorbed}\ {
m ray}.$  For the photodecomposition of the undrawn fibre, the activation energy is about 14 kcal./mole, which agrees with the value obtained from the thermal decomposition of the same fibre. The following relation between the volume x of hydrogen chloride liberated and the time t is obtained

$$kt^{\frac{1}{m}} = \ln\left(\frac{a}{a-x}\right)$$

(k and m are constants; a = initial polymer content).

### Effect of Molecular Weight and Molecular-weight Distribution on the Physicomechanical Properties of Khlorin Fibre [Chlorinated Polyvinyl Chloride] B. É. Geller

Tekstil. prom., 16, 14-16 (Nov. 1956) For the preparation of Khlorin fibre having satisfactory physicomechanical properties the polymer used must have a D.P. of 900–950 and must contain not more than 8-10%of fractions of D.P. <450 and not more than  $2\text{-}3\,^\circ_{\scriptscriptstyle 0}$  of organic impurities. The use of polymer having low contents of low-mol.wt. fractions and organic impurities results in a fibre of high thermal stability.

### PATENTS

### Saponification of Cellulose Acetate

Filaments are formed from cellulose acetate of acetyl value (calc, as acetic acid) > 58% and then after being freed from solvent but not dried are treated with a mineral alkali or ammonia or an amine in a liquid containing no swelling agent or solvent for the cellulose acetate, until the cellulose acetate has an acetyl value of 42-52%. Under these conditions the acetyl value of the filaments is a linear function of the time of treatment. In a modification of the process material which has been dried is similarly saponified by using a weak base, e.g. ammonia or an amine. When the resulting filaments are dyed with

direct or "plastosoluble" dyes their cross-sections show homogeneous coloration. C.O.C.

### Cellulose-Phenylthiourethan Fibres

USP 2,728,630 American Viscose Corpn. USP 2,728,630 Fibres having a dry extensibility of  $\langle 15^{\circ}_{\circ \circ}$ , resiliency,

and good wet and dry tenacities are obtained by extruding an aqueous alkaline solution of cellulose-phenylthiourethan into an aqueous coagulating bath of pH 3-2-6-2 and of a specified dehydration value (special test described). They have a crenellated cross-section and have high waterretention capacity coupled with low cross-sectional and linear swelling in presence of water. They are very suitable for blending with wool and have affinity for wool

### Polyacrylonitrile Solutions

BP 768,708 Courtaulds

Polymers containing < 80% by wt. of acrylonitrile are dissolved in a solution of zinc and calcium chlorides in water containing a water-miscible aliphatic liquid of  $<7\,\mathrm{C}$  containing an alcoholic hydroxyl group. The salts form <35% by wt. of the solvent mixture. Filaments are formed by extruding such a solution into water,

### Dextran Ether Fibres

Commonwealth Engineering Co. of Ohio A water-insoluble dextran ether and cellulose nitrate are dissolved in a common solvent, e.g. Cellosolve, and then dry-spun.

### Acrylonitrile Polymer and Copolymer Filaments

Filaments made from a copolymer of acrylonitrile (90-70%) and vinyl acetate (10-30%) have good affinity for most classes of dyes,

## Viscoelastic Properties of Textile Fibres. 11—Influence of Physical Treatments on the Dynamic Properties of

some Fibre-forming Polymers (X p. 184) Improving the Dimensional Stability, Wet Strength, etc.

of Cellulosic Textiles (X p. 184) Elimination of Greasy Impurities from Waste Paper

before Re-use (XI p. 185)
Influence of Aqueous Hydrolysis of Pine Wood on Cellulose produced from it by the Sulphate Method (XI p. 185) Acid Hydrolysis of Acrylonitrile Copolymers (XIII p. 186) Pigmented Organic Plastics (XIII p. 186)

### VII-DESIZING; SCOURING; CARBONISING: BLEACHING

### New Applications of Starch Esters and Ethers in the Detergent Field

### J. Paliot

World Congress on Surface-active Agents, 1, 352 357 (1954)

The products (Solvitoses) are claimed to protect against wear and to prevent redeposition of soil in laundering, to enhance emulsifying power, and to stabilise foam, and are also used for thickening liquid detergents. They are discussed under these headings, S.R.C.

### Catalytic Fibre Damage in Bleaching

W. Schönberger World Congress on Surface active Agents, 2, 462–470 (1954)

462-470 (1954)

The effects of H<sub>2</sub>O<sub>4</sub>, its derivatives, and hypochlorite on cellulosic fibres dyed with Cu-, Co-, Cr-, and Fe-containing dyes, e.g. phthalocyanines and metallised direct dyes, were examined using rip-strength as a measure. The dyes can cause damage depending on the nature of the metal and its content in the molecule. The type of combination between metal and colouring matter is of prime importance. Viscose rayon staple is more sensitive to catalytic damage than is cotton.

### Collated Results of Studies on Cotton Piece Scouring

World Congress on Surface active Agents, 2, 478-486 (1954)

The use of > 2 g. Na<sub>3</sub>CO<sub>3</sub> per litre is wasteful and may even reduce the effectiveness of the scour. In hard water, the alkaline salt should be added before the soap, but additions of soda permit economies in soap concentration even in soft water. If the water contains Mg or bicarbonates, Na<sub>2</sub>PO<sub>4</sub> is preferable to Na<sub>2</sub>CO<sub>2</sub>. S.R.C.

Non-ionic Surfactants in Raw-wool Scouring M. M. Thiry

World Congress on Surface-active Agents, 2, 429-432 (1954)

Laboratory evaluation of condensates of ethylene oxide with (1) lauryl alcohol, (2) a fatty-acid amide, (3) octylphenol, (4) nonylphenol showed (4) to be superior. This product was then examined on the semi-large scale with good results. For grease-recovery, centrifuge methods are claimed to be effective with non-ionic S.R.C.

PATENTS

Preventing Chemical Degradation of Cellulose during Washing BP 765,215

Kraemer & Flammer Salts of a sulphonated or sulphated synthetic organic detergent with a metal of Group II or III of the Periodic System, e.g. Al, form aqueous dispersions containing negatively charged particles which have an oxygen-stabilising effect. Added to detergents, particularly those containing per-compounds, they greatly reduce the tendency for cellulose being washed with such detergents to become degraded.

Washing Natural or Synthetic Fibres

RP 764-007 Boehme Fettchemie

If during washing the fibres and dirt are positively charged by using at > 50°c, a wash liquor containing at least 0.85 g. of positively charging substance per litre, at least a part of which is a cationic detergent, good cleansing of both natural and synthetic fibres is obtained. Cleansing is improved if "cationic colloids", i.e. colloids which promote the positive electrical charging of the fibres and dirt, are simultaneously used. Thus white nylon is cleansed perfectly in an aqueous liquor at 40-50°c, containing per litre 0.4 g. of octadecoxy-carboxymethyl-cyclohexyldimethylammonium chloride, I g. of acetic acid and 0.03 g. of methylcellulose.

Wool Scouring

RP 766,762 John Stevens Pty.

Wool which has been deburred, and as much dirt as possible removed mechanically, is solvent-extracted, preferably with a non-ionic low-boiling petroleum solvent, e.g. hexane, until no more wool fat can be extracted. is then treated with an aqueous solution of a non-ionic detergent at room temperature, rinsed and dried. method yields a high-quality product and enables all the by-products to be recovered in relatively pure form.

Soaps and Sequestering Agents—Complete Detergents (II p. 174)

Non-ionic Products in the Treatment of Hydrophobic

Fibres (III p. 174) Use of Fluorescent Brightening Agents in the Detergent Industry (III p. 174)

### VIII- DYEING

**Infrared Reflection of Coloured Textiles** 

E. Fritsche and M. Kracht

Textil-Praxis, 12, 58-61 (Jan. 1957)

The significance of the infrared reflection of textile materials, particularly for camouflage work and the selection of coloured clothing for wear in warm climates, is discussed.

Surface-active Compounds as Retarding Agents in the Dyeing of Cellulosic Fibres

P. Flamant

World Congress on Surface-active Agents, 2. 495-498 (1954)

All surfactants capable of forming labile micellar systems with dye ions should give aggregates which liberate the dye progressively and completely with rise in temperature or other change in conditions. Ampholytic retarding agents are mixtures of anionic and cationic products, e.g. heptadecyl-N-ethylmethylbenziminazolium chloride + sodium N-methylheptadecylbenziminazole-sulphonate. This gives a feebly dominant cationic mixture capable of retarding anionic dyes at initial dyebath

temperatures and slowly releasing dye as the bath warms

Diffusion of Direct Dyes into Cellulose
B. N. Mel'nikov and P. V. Moryganov

Colloid J. U.S.S.R., 18, 566-573 (Sept.-Oct. 1956)

Diffusion coefficients of four direct dyes are studied, and the effects of change of temp, (over the range 40-100°C.) and of electrolyte conen. (0·1-15 g./litre) are related to the energy of activation. The results are explained on the basis of gradual (electrolyte effect) and drastic (temp. effect) changes in the degree of dispersion.

Sorption of Dyes by Fibres in a Vacuum

S. M. Lipatov and N. D. Ilyashuk Colloid J. U.S.S.R., 18, 562-565 (Sept.-Oct. 1956) The solubility of Direct Brown KKh is studied at low temp. and the formation of stable supersaturated solutions (due to aggregation) is shown. As compared with normal conditions, the dyeing of cotton (25-40°c.) with Direct Brown KKh (C.I. Direct Brown 2). Direct Violet (C.I. Direct Violet 1), and Chrysophenine (C.I. Direct Yellow 12) (as measured by the time to reach equilibrium) is considerably less when applied in vacuum (10 mm. Hg). The influence of NaCl is investigated: addition of 15% increases dye uptake from 74% (no salt) to 84%; 32% NaCl decreases the dye uptake to 61%. G.J.K.

Chemistry of Esters of Leuco Vat Dyes. II-A Comparison of the Rates of Oxidation with the Standard Redox Potentials of the Parent Vat Dyes

S. Ainsworth and A. Johnson J.S.D.C., 73, 41-46 (Feb. 1957)

The rates of oxidation, with acidic hydrogen peroxide, of a number of disulphuric esters of leuco vat dyes have been measured and compared with the standard redox potentials of the parent vat dye-leuco vat dye systems. On the basis of this comparison the esters may be divided on the basis of this configuration of the relative importance of oxidation or hydrolysis in the development sequence.

AUTHORS

Астнов

Application of Highly Dispersed Vat Dyes

A. Schlottmann

Textil-Praxis, 12, 62-69 (Jan. 1957) The following brands of highly dispersed anthraquinone dyes are marketed on the Continent-Colloisol (BASF), Microdisperse (Ciba), Supra (FH), Superfine (Fran), and Ultrafein (FBy). The advantages of the Ultrafein range in package dyeing, piece dyeing, dope dyeing, and continuous dyeing processes are described. A number of colour photographs illustrate the state of dispersion and advantages in dyeing possessed by the Ultrafein dyes compared with the normal quality.

B.K. Dyeing Properties of Disperse Dyes in an Acid Bath Y. Tanaka

J. Soc. Textile Cellulose Ind. Japan, 12,

The change of colour and F-value (Kubelka-Munk equation) of dyeings of cellulose acetate dyed from baths acidified with acetic or sulphuric acid (0.005-0.5 g. per 100 c.c.) were measured by means of a spectrophotometer, and the results are expressed on the C.I.E. system. Nine disperse dyes of the azo or anthraquinone series were used, and nine experiments were made with neutral dyebaths for each dye. It was found that generally the change in dominant wavelength was small, and the excitation purity increases in the CH2 COOH bath but decreases in the H<sub>2</sub>SO<sub>4</sub> bath for most dyes. The F-value for all the dyes examined increases in the acid bath; it increases in low conen. of H<sub>2</sub>SO<sub>4</sub>, while it decreases as

Dyeing of Woolskins

the H<sub>2</sub>SO<sub>4</sub> conen. increases.

Leder u. Hautemarkt tech. Beilage, 56 (1956): J. Soc. Leather Trades Chem., 40, 409 (Dec. 1956)

In a general review of the uses and properties of anionic dyes, it is stated that in the dyeing of woolskins temperature is of great importance. Where it is desired to dye the leather and not the wool, a dye of high affinity for chrome leather should be used at 25°c. If such a skin is then placed in a float at 60°c., the dye will migrate from the leather to the wool. C.J.W.H.

### Dyeing of Wool, Hair, and Polyamide Fibres with Metal-complex Dyes

E. Schönpflug

Textil-Praxis, 12, 70-79 (Jan. 1957)

A review of the chemistry of 1:1 and 1:2 metal complexes and their application in the dyeing of wool, fur felt hoods, polyamide fibres, and unions of wool and polyamide

### Mass Coloration of Capron [6-Nylon]

S. Z. Baibuim

Tekstil. prom., 16, 19-20 (Sept. 1956)

Capron crumbs are dusted with vat dyes of particle size  $\gg 1\,\mu$ , or are dyed from an alkaline vat. In the latter case some low-mol.wt. polymer is leached out and the spinning characteristics of the melt are altered. The coloured Capron fibre obtained from the crumbs must be stretched without delay, for it is found that drawability diminishes rapidly with time. So long as these factors are taken into account, the quality of the fibre is not affected by mass coloration.

# Dyeing of Difficultly Dyeing Fibres. IV— Condensa-tion-dyeing by Active Halogeno Compounds with Free Amines (1)

K. Yamada

J. Soc. Textile Cellulose Ind. Japan, 12, 834-839 (Nov. 1956)

Active halogeno compounds, e.g. picryl chloride, readily react with various free amines to give fast and level dyeings on the several kinds of fibres. Amines of the benzidine series were excellent, and basic dyes having a free amino radical in their molecules, which combines with picryl chloride, increase the fight fastness. Uniform colour in the crystalline regions of Vinylon, and avoidance

of spots or colour removal from various fibres were made more satisfactory by suitable selection of dyeing process. The order of dyeability of the fibres concerned is as follows: vinyl chloride film > cellulose acetate > Vinylon and

cotton > silk. The dyeing mechanism of these fibres appears to be of the solid-solution type. AUTHOR

Acrylics and Dyers

J. Corbière

Teintex, 22, 17-32 (Jan. 1957)

Some physical characteristics of Orlon, Acrilan, Creslan, Darlan, Crylor, Dralon, Redon, and Dolan are tabulated. For Crylor the preparation, setting (for which saturated steam at 3 kg. pressure, i.e. 135 c., for 15-45 min., water boiling at 135°c., or dry setting at 180-190°c. is recommended), bleaching (3 g. sodium chlorite plus 1 g. ethyl lactate or tartrate per litre, together with sodium nitrate (1.5 g./litre) if it is required to ensure that stainless steel is not corroded), and dyeing with basic dyes (pH = 4 with CH<sub>3</sub>COOH plus CH<sub>3</sub> COONa (0·15 g./litre) for increasing fastness to ironing, hot-pressing, or post-setting + wetting agent), "plastosoluble" dyes (formulae given), and acid dyes with hydroxylamine-Cu or hydrazine-Cu at 100°c., NaHSO\_Cu above 100°c., or the Sandocryl process at or above 100°c. are discussed. The dyeing of Crylor unions is reviewed.

Acrylic Fibres- Dyeability

M. Katayama and K. Sakaba J. Soc. Textile Cellulose Ind. Japan, 12.

809-814 (Nov. 1956)

Determination of the absorption of acid, basic, and disperse dyes by pure polyacrylonitrile fibre and acrylamide copolymer fibres showed that—(1) dyeability to basic dyes is increased by copolymerisation with acrylamide; (2) primary and secondary amino groups in the dye molecule increase the absorption; and (3) acrylamide copolymer fibre treated with HCl shows excellent dyeability to acid dyes, but is inferior to the other dyed fibres in wash fastness.

PATENT

Dyeing or Printing Textiles containing Cellulose

A neutral to alkaline dyebath or printing paste is used containing an amine having at least one amino group separated from a hydroxyl group by 2 C, a compound containing anions of a phosphoric acid, an alkali-resistant agent yielding Cu, and a dye free from complex-forming metal but capable of forming a sparingly soluble Cu complex and containing > 1 azo linkage and o-hydroxycarboxylic acid groups as its only metallisable groups, The dye must be such that its copper compound is 90% soluble when boiled in 50 times its wt. of water in presence of twice its wt. of 1:2-di(β-hydroxyethylamino)-ethane, Thus I part of the dye obtained by treating I mol. of-

and 1 mol. of-

with phosgene is dissolved in water (200). 100 Parts of the resulting soln. are added to water (2000) at 60°c, 50 Parts by volume of a solution of sodium copper pyrophosphate (1) and 1:2-di-( $\beta$ -hydroxyethylamino)-ethane (2) in water (100) are added followed by wetted-out cotton (100). The bath is brought to the boil in 30 min., boiled for 1 hr. during which Glauber's salt (15 and 15) is added after 5 and 20 min, respectively. This yields a yellow dyeing of good fastness to light and washing. C.O.C.

Non-ionic Products in the Treatment of Hydrophobic Fibres (III p. 174)
Use of Fluorescent Brightening Agents in the Detergent

Industry (III p. 174) Dyes for Polyvinyl-derivative Fibres (IV p. 178) Progress in Man-made Protein Fibres (VI p. 180)

Acrylic Fibres. XVII - Binary and Ternary Copolymers of Acrylonitrile (VI p. 181)

Saponification of Cellulose Acetate (VI p. 181) Printing and Padding of Aerylonitrile Mixed Polymers (IX below)

Auxiliary Products as Fat Liquors in the Dyeing of Suede and Gloving Leathers (XII p. 185)

### IX-PRINTING

Origin and Early Development of Textile Printing in Switzerland, especially in the Canton of Glarus U. F. Blumer

Textil-Rund., 12, 8-20 (Jan. 1957)

Printing and Padding of Acrylonitrile Mixed Polymers

Textil-Praxis, 12, 80 (Jan. 1957)

Lanasyn (8) metal-complex dyes give prints on dynel of high fastness to light. The dyes are applied in cone, form together with an organic solvent (in particular mixtures of phenols and alcohols), a thickener, and thioures. After printing dry at 50 c., steam for 30 min. at normal pressure, rinse in cold water, soap with a sulphated fatty alcohol at 50-60 c., rinse, and dry.

### Emulsification of Offset Inks

J. H. Bitter

Textil-Rund., 12, 24-29 (Jan. 1957)

Good results in offset printing on coated paper are obtained only if the lowering of the surface tension of the water by the paper does not exceed a specific maximum. The pH of the repellent water must lie between 5 and 6. higher values causing flooding and lower values corroding zinc plates. No surfactant must contaminate the repellent

Influence of Gelatin on Optical Sensitivity of Photographic Emulsions

A. V. Borin and V. P. Agatitskaya J. Appl. Chem. U.S.S.R., 29, 1393-1401 (Sept. 1956) When cone, gelatin soln, (of the order of 5–10%) are coloured by a dye which reacts chemically with gelatin or which is adsorbed by it, the intensity of the j-band can vary from almost complete absence to a high value. The distribution of the spectral sensitivity of an emulsion sensitised by such a dye depends upon the type of gelatin in the emulsion. The relative intensities of the fundamental absorption bands of the coloured gelatin solutions agree well with the relative intensities of the corresponding sensitisation bands. It is assumed that the influence of

the gelatin on the spectral sensitivity is caused by the adsorption of the dye in the form of a dye-gelatin complex, which has a fixed spectral absorption. When emulsions are sensitised by a dye which does not react with gelatin, the distribution of spectral sensitivity is determined mainly by the state of the dye in the adsorption layer on the surface of the microcrystals of AgBr. Gelatin exerts a retarding influence on the aggregation of the dye in solution, which causes an increase in concn. of the molecular form of the dye, and this in turn favours the formation of a "mesophase" of the dye, on the surface of the AgBr. In such a case, if gelatin is present in the emulsion, forming the mesophase of the dye in the soln., the conen, of the molecular form of the dye decreases, and consequently the concentration of the dye mesophase on the surface of the AgBr decreases. This causes a decrease in the spectral sensitivity of the emulsion in the vicinity of the j and a absorption bands. T.Z.W.

PATENTS

### Colour Photography

BP 768,707 In colour-photographic material at least one of the lightsensitive layers comprises a dispersion of a water-insoluble or sparingly soluble di- or tri-substituted guanidine salt of a colour former containing a sulphonic or carboxylic acid group and a hydrocarbon chain of > 4 C in a gelatin- or gelatin substitute silver halide emulsion. C.O.C.

Ascorbic Esters as Antistain Agents in Colour Photography Eastman Kodak Co. USP 2,728,661

N-Alkylquinols as Antistain Agents in Colour Photography USP 2,728,659

Dyeing or Printing Textiles containing Cellulose (VIII p. 183)

### X-SIZING AND FINISHING

Polyvinyl Alcohol as a Warp Size for Various Staple

E. Abrams, C. W. Rougeux, and J. N. Coker

Text. Research J., 26, 875-880 (Nov. 1956)
A completely hydrolysed, high-viscosity polyvinyl alcohol, tested under simulated weaving conditions, was found to be an effective size for cotton, Dacron, and blends of Dacron with cotton and viscose rayon. The same product was also effective as an extender for starch-based

Viscoelastic Properties of Textile Fibres, II-Influence of Physical Treatments on the Dynamic

Properties of some Fibre-forming Polymers
K. Fujino, H. Kawai, T. Horino, and K. Miyamoto

Text. Research J., 26, 852-871 (Nov. 1956)

The relaxation spectra of the typical fibre-forming
polymers polyethylene terephthalate (I), polycaproamide (II), polyvinyl chloride (III), and polyvinyl alcohol (IV) have been investigated over the frequency range 2 × 10-1 to 2 × 105 cycles/sec., and the effects of cold drawing and heat treatments on the spectra studied. Cold drawing of I and III increases the relaxation spectrum almost formly over the whole range, whilst with II and IV the spectrum increases only on the longer-relaxation-time side. Heat treatment of I has little effect on the spectrum, despite the greater degree of crystallisation, whilst for II and IV the spectrum is decreased on the shorter-relaxationtime side and increased on the longer-relaxation-time side as the treatment progresses. The results are discussed in in the light of structural concepts. J.C.F.

Application of Crease-resistant Finishes to Cotton A. R. Smith

Text. Research J., 26, 836-851 (Nov. 1956) Crease-resistant finishes based on a series of ureaformaldehyde and melamine-formaldehyde precondensates were applied to cotton fabric. Treated samples were tested for crease resistance, tensile strength, and tear strength. Certain comparative experiments were made The following general conusing viscose rayon fabric. clusions emerged - (a) for all types of precondensates the reduction in strength is proportional to the orease resistance imparted; (b) the order of efficiency for a given resin concentration is: ethyleneurea-formaldehyde

urea-formaldehyde > melamine-formaldehyde; (c) the degree of condensation of the precondensate has little effect on finishes on cotton, whereas on viscose rayon the more highly condensed products have an adverse effect on handle and crease recovery; (d) washing off improves the tear strength of treated cotton but adversely affects the tensile strength; (e) thermoplastic additives improve tensile strength to some extent, but have an adverse effect on tear strength; (f) softeners have a similar effect to washing off; (g) mercerisation of cotton has a beneficial effect on its behaviour after finishing; and (b) fabric construction has an important effect on the strength of the finished fabric. The mechanism of crease resistance is discussed.

### Surfactants in the Rubberising of Textiles

World Congress on Surface-active Agents, 3, 1175-1177 (1954)

Soaps assist in insolubilising Mn, Fe, Cu. mixtures contain wetting, foaming, dispersing, stabilising, or emulsifying agents. The adhesion of rubber to textiles is enhanced by pretreatment of the latter with cationics. S.R.C.

Rabbit Fibres

#### XII - Mechanical Properties of Carrotted Fibres -Effect of Immersion Temperature and Drying Conditions

S. Okajima, S. Ikeda, and A. Takezawa

J. Soc. Textile Cellulose Ind. Japan, 12,

790-795 (Nov. 1956)

Carrotting conditions for the Angora rabbit guard hairs were studied by tracing the change in mechanical properties. The carrotting agents contained 1.8% Hg(NO<sub>2</sub>)<sub>2</sub> and 3.6% HNO<sub>2</sub>. The immersion was carried out at 20, 30, 45, and 60°c. The drying of the hairs after immersion was and 60°c. The drying of the hairs after immersion was made for 2 days at room temperature or for 5, 15, 30, 60, and 120 min. at 95°c, respectively. It was observed that the mechanical properties of the hairs were modified remarkably by raising the immersion temperature, but the effects of the drying temperature and time were negligible.

### XIII- Mechanical Properties of Carrotted Fibres — Effect of the Immersion Conditions S. Ikeda, S. Okajima, and S. Kato

Angora rabbit fibres were immersed in solutions containing various amounts of Hg(NO<sub>s</sub>)<sub>2</sub> and HNO<sub>s</sub> for various lengths of time at 30°c, and dried for 30 min, at The mechanical properties of the treated fibres were tested, and the following results were obtained-(1) The effects of the time of immersion (5-65 min.) upon the 30%-extension and the work of recovery are significant at the 1% level, and the effect upon the resilience is also significant at the 5% level. (2) The effects of the concn. of  $Hg(NO_2)_4$  (0.5–3.5%) upon the extension and the work of recovery are significant at the 5% level. (3) The concn. of HNO<sub>3</sub> (1.5-4.5%) does not affect significantly any of the above parameters at the 5% level.

Then another series of experiments was carried out in which the composition of the carrotting liquor was kept constant  $(0.5\% \text{ Hg(NO}_3)_5 + 1.0\% \text{ HNO}_3)$  and the time and the temperature of immersion were changed. The 30% extension and the work of recovery decreased to 60-73% and 56-72% of the original values on immersion for 2 hr. at 60°c. AUTHORS

PATENTS

### Imparting to Continuous-filament Yarn the Appearance and the Handle of Staple-fibre Yarn

BP 767.835 Algemene Kunstzijde Unie Twist is imparted to the yarn after which it is forced through a confined zone by a high-velocity gas stream. On leaving the confined zone the direction of travel of the yarn is changed and the speed reduced without applying further twist. This results in loops being formed at random in the individual filaments and so imparts a springiness which cannot be achieved by the usual crimping or twisting proce

### Improving the Dimensional Stability, Wet Strength, etc. of Cellulosic Textiles

American Enka Corpn. USP 2,728,628 The materials are treated with a weakly acidic dilute solution of a long-chain dialdehyde. Thus viscose rayon

yarn while still wet and in the gel state from formation is treated for 5 min. at 50-60 c. in an aqueous solution containing 8% a-hydroxyadipaldehyde, dioxan (to prevent precipitation on dilution) and oxalic acid (as catalyst), centrifuged for 5 min., dried for 1 hr. at 50-55°C, and finally baked.

Reducing the Felting Power of Wool

Dow Corning Corporation USP 2,728,692 Wool fabric is treated with a composition consisting of (1) 25-95% by wt. of a benzene-soluble diorganopolysiloxane of viscosity « 1 million centipoises at 25°c, and (2) 75-5% by wt. of a compound of formula-

 $R_n \mathrm{SiH}_m \mathrm{O}_{(4-m-n)/2}$ 

(R = Alk, alkenyl or phenyl; n averages  $1 \cdot 0 \cdot 1 \cdot 0$ ; m averages  $0 \cdot 1 \cdot 1 \cdot 0$ ;  $m + n = 1 \cdot 65 \cdot 2 \cdot 1$ ), and baked, e.g. for 10 min.

Fabrics coated with Thermoplastic Synthetic Resin Bernard Wardle (Everflex)

Fabric coated with a synthetic thermoplastic resin is passed through water at 75–100°c, and dried in absence of longitudinal tension. The treated fabric can be affixed to curved surfaces without creasing. C.O.C.

Improving the Abrasion Resistance of Textiles E. X. C. M. A. Dupont BP 765,735

A lubricant is added to the rinsing bath and if desired emulsified non-plasticised synthetic resin, polyvinyl acetate, to stiffen the goods.

Alkali-metal Borohydrides as Hair-waving Compositions

(III p. 175)
Fault in Viscose Rayon-Cellulose Acetate Fabric due to
Action of Light during Storage (VI p. 180)

### XI-PAPER AND OTHER CELLULOSIC **PRODUCTS**

Elimination of Greasy Impurities from Waste Paper before Re-use

N. Vaucher

World Congress on Surface-active Agents, 2. 606-609 (1954)

The impurities are (1) paraffin, (2) microcrystalline waxes, (3) resins, and (4) tar. Xylene is the best solvent for (4), trichloroethylene for (1)-(3). By emulsifying with a surfactant and employing 1% of mixed solvent calculated on the water, good removal is effected. The paper should be entered dry.

Influence of Aqueous Hydrolysis of Pine Wood on Cellulose produced from it by the Sulphate Method F. I. Korchemkin

J. Appl. Chem. U.S.S.R., 29, 1440-1442 (Sept. 1956) The cellulose produced from pine wood by the sulphate method shows fibres having damaged outer layers T.Z.W.

Distribution of Substituents in Hydroxyethylcellulose

I. Croon and B. Lindberg

Svensk Papperstidning, 59, 794-799 (30 Nov. 1956) Hydroxyethylcellulose (0-60 C<sub>2</sub>H<sub>4</sub>-O per glucose unit) was hydrolysed to monomers, which were fractionated on a carbon column. Glucose and three monosubstituted and six disubstituted ethers were isolated and charac-terised. The relative amounts of the monomers were in good agreement with the calculated values assuming that all glucose units are equally accessible and that the relative velocity constants for etherification of the hydroxyls at  $C_{(a)}$ ,  $C_{(a)}$ ,  $C_{(a)}$ , and the hydroxyethyl groups are 3:1:10:20. About 25% of the ethylene oxide units introduced had reacted with the hydroxyethyl groups to form polyethylene oxide chains.

PATENTS

Coloured Cellulose Ethers and Xanthates

Manufactures de Produits Chimiques du Nord Établissements Kuhlmann BP 766,198

Cellulose is dyed in any suitable manner, washed, dried, converted into alkali cellulose with aq. NaOH, shredded and then converted to a cellulose ether or xanthate. The xanthate so produced can be used in the normal manner to produce coloured filaments. The water-soluble coloured cellulose ethers produced may be used for tinting and blueing pastes in papermaking, water paints, distempers,

Paper for Making Copies

Standard Register Co. A sheet of paper to be written or typed, etc. on one side is coated on the other side with a water-soluble metal salt which gives a dark colour with H<sub>2</sub>S, a water-insoluble polyvalent-metal salt of a higher carboxylic acid which does not react with H<sub>2</sub>S, a light-coloured natural or synthetic resin, and a volatile organic liquid such as C4H4 or C4H3CH3. The coating is transferred to another sheet in contact with it and rendered legible by treatment with dry H2S gas.

Pressure-sensitive Paper

Caribonum

A base sheet is printed or coated with colouring materials that are fast to light and of suitable grain. A white top coating of precipitated CaCO<sub>2</sub> dispersed in a binder (sodium alginate, gum arabic, glue, etc.) is then applied. The white coating is rendered transparent when an oil or oily substance comes into contact with it, e.g. by means of an oil-impregnated typewriter ribbon, or the oil may be applied to the coloured paper surface and protectively covered with a layer of a water-soluble cellulose derivative, to which the white coating is finally applied. The protective layer is ruptured by writing or typing action. the oil is applied to the reverse side of the coated paper, the treated sheets may serve as copy paper. Colouring of the base sheet is unnecessary if the CaCO<sub>3</sub> is mixed with 10% or less of colouring material prior to coating.

### XII- LEATHER; FURS; OTHER PROTEIN MATERIALS

Chemical Studies on the Liming of Animal Skin. VI-Effects of Organic Nitrogen Compounds in Accelerating the Unhairing of Animal Skin by Lime Liquor. I-Amines

H. Toyoda and A. Fukami

Rept. Govt. Chem. Ind. Rev. Inst. Tokyo, 51. 429-436 (Nov. 1956)

The activity of amines in accelerating the unhairing of animal skin is closely related to their chemical structure and is not always parallel to their strength or basicity. Thus, it may be concluded—(1) that primary and second-ary aliphatic amines have an accelerating action, but tertiary aliphatic amines and aromatic amines have no effects; and (2) that when negative groups such as acetyl and phenyl are introduced into the molecules of amines, the accelerating power of those amines is markedly decreased or completely lost.

AUTHORS

Auxiliary Products as Fat Liquors in the Dyeing of Suède and Gloving Leathers

C. Faure and H. Poinsard
World Congress on Surface-active Agents, 2, 572-578 (1954)

Anionics reduce the colour yield of direct and acid dyes, particularly those of low mol. wt. or of high sulphonation. Cationics increase the colour yield of anionic dyes and are of interest for medium to deep colours, but penetration is limited. Non-ionics give the most satisfactory results for levelling of pale dyeings, penetration, and colour yield of anionic dyes. S.R.C.

Investigations into the Behaviour of Auxiliary Materials in Leather Manufacture by means of Radioactive Substances. I- Effect of Surface-active Agents

R. Heyden, J. Plapper, and K. Sauerwein

Leder, 7, 100 (1956): J. Soc. Leather Trades Chem., 40, 410 (Dec. 1956)

The behaviour of wetting, dispersing, and emulsifying materials was studied by employing those containing 118. It was found that these were bound to the hide protein, the degree of binding being the greater the later they are applied in the leather-manufacturing process. It has been stated that the water uptake of leather is increased by the use of surface-active agents in its manufacture: it was found that anionic agents had no appreciable effect on

water uptake, but the conditions of leather manufacture influence the effect. The use of excessive amounts can increase water uptake. It is found that anionic agents collect under the grain and in the pores, which may explain the satisfactory effects of such agents on liming, scudding, and the quality of grain. With small additions of surfaceactive agents, almost all is attached to the pelt surface; increasing amounts, particularly with a short float, will produce penetration. The use of anionic agents of low molecular weight in vegetable tannage is claimed to increase rate of penetration, to protect the grain, to equalise tan distribution, and to improve tensile strength. C.J.W.H.

### PATENT

### Organosilicon Compositions containing Zirconium for treating Leather

Dow Corning Corpn.  $USP\ 2,728,736$ A mixture of (1) 15–50% by wt. of a compound of formula  $Zr(OR^3)_4$  or aliphatic-hydrocarbon-soluble partial hydrolysates ( $R^3$  = aliphatic hydrocarbon of < 13 C and may contain 1-3 OH groups), (2) 5-70% of a methyl-polysiloxane resin in which the ratio of CH<sub>8</sub> to Si is 1:1 to 2-5:1, and (3) 15-80% by wt. of a polysiloxane of

$$\mathrm{R^2}_n\mathrm{SiO}(4-n)/2$$

(R2 = Alk or alkenyl of < 6 C or monocyclic Ar; n averages 2.0-2.9) is used to render leather water-repellent and resistant to wear and weathering.

Dyeing of Woolskins (VIII p. 182)

Detection of Traces of Formaldehyde in Dried Hide and

Skin Material (XIV p. 188)
Thiohydantoin Method for the Determination of the Terminal Carboxyl Amino Acids of Collagen (XIV p. 188)

### XIII- RUBBER; RESINS; PLASTICS

### Sorption of Water Vapour by Water-soluble Polymers

L. J. T. Hughes and D. B. Fordyce

J. Polymer Sci., 22, 509-526 (Dec. 1956) Equilibrium and kinetic studies of sorption and desorption of water vapour by poly(methyl acrylate) hydrolysates, poly(acrylic acid), poly(sodium acrylate), sodium carboxymethylcellulose, and methylcellulose are reported. Equilibrium sorption increases and is greater for polymers containing strong-electrolyte-type sub-stituents than for those with weakly ionisable units. Diffusion is somewhat slower than with water-insoluble polymers. Anomalous diffusion and relatively high glass temperatures are associated with high concentrations of very polar or ionic constituents.

### Kinetics and Mechanism of Thermal Decomposition of Chlorine-containing Vinyl Polymers N. V. Mikhailov, L. G. Tokareva, and V. S. Klimenkov

### I- Technique and Results for Polyvinyl Chloride

Colloid J. U.S.S.R., 18, 578-582 (Sept.-Oct. 1956) The amount of Cl eliminated as HCl and the residual Cl when polyvinyl chloride (P.V.C.) is decomposed under N, at 150-170 c. are determined. A formal zero-order reaction is claimed, and the energy of activation calculated to be 36.5 kcal./mole. Solubility and viscosity measurements show that on heating P.V.C. polymerisation and degradation occur simultaneously.

# II—Results for Polyvinylidene Chloride, for a Copolymer of Vinylidene Chloride and Vinyl Chloride, and for Chlorinated Polyvinyl Chloride

The effect of oxygen on the thermal decomposition of further chlorinated polymers is to accelerate the decomposition process. It is suggested that decomposition has a radical-chain mechanism, e.g.-

G.I.K.

### Acid Hydrolysis of Acrylonitrile Copolymers

G. I. Kudryavtsev and M. A. Zharkova J. Appl. Chem. U.S.S.R., 29, 1103–1108 (July 1956) It is shown that I:1 copolymers of acrylonitrile with methyl acrylate, vinylidene chloride, methyl methacrylate, vinyl chloride, and styrene are progressively less difficult to hydrolyse with acid, and all of them are less difficult to hydrolyse than polyacrylonitrile. This sequence corresponds closely with that to be expected from the magnitudes of the inductive effects produced in the CN groups by the side-chains introduced by copolymerisation.

#### PATENTS

### Incorporating Plasticisers into Aqueous Dispersions of Polymers

BP 766,654 Emulsified plasticisers are incorporated into aqueous dispersions of polymers by adding them at 80-90 c.

C.O.C.

### Pigmented Organic Plastics

#### B. F. Goodrich Co. BP 768,844

The plastic and the pigment are treated in a ball, pebble or colloid mill in presence of a mixture of water and two organic solvents for the plastic, the one of which the larger quantity is used being miscible with water and the other immiscible. This yields a fine, stable dispersion, which can be used as a master colour batch. Because of the fineness of the dispersion plastics so coloured before being made into filaments yield filaments of the same tensile strength as similar unpigmented filaments.

#### Patterns resembling Watermarks in Foils of Polyvinyl Chloride and Similar Plastics BP 766,181

When the foil is passed over a heated roller before being stretched effects resembling watermarks are produced if the roller has a pattern in low relief on it, e.g. if it is etched. Very delicate patterns may be obtained, and the resulting foil has the same mechanical properties as if the roller had been completely smooth and polished. COC

### Aromatic Diamines for preventing Bloom on

#### Rubber-Resin Blends United States Rubber Co.

Dispersing aromatic amines, e.g. p-phenylenediamine, added to blends of styrene-acrylonitrile resin and butadieneacrylonitrile rubber, prevent the blend from blooming.

### Thickening Aqueous Dispersions of Polymers of Alkoxyalkyl Methacrylates (V p. 180) Surfactants in the Rubberising of Textiles (X p. 184)

### XIV— ANALYSIS; TESTING; APPARATUS

### Cone-Plate Viscometer

R. McKennell

Anal. Chem., 28, 1710-1714 (Nov. 1956)
A review of the theory of the cone-plate viscometer is given, and a comparison is made with the coaxial-cylinder viscometer.

### Estimation of Butyl Alcohol and Cellosolve in Effluents with Sudan III Reagent

D. H. Feldman and J. C. Cavagnol Anal. Chem., 28, 1746–1748 (Nov. 1956) Butyl alcohol or Cellosolve may be determined in dil. aq. mixtures by use of a reagent consisting of solid Sudan III (C.I. Solvent Red 23), NaCl. and K<sub>2</sub>CO<sub>2</sub>, the colour change and extent of extraction of the dye being compared with standards. The method is applicable to aq. mixtures of the two solvents where the conen. of each does not exceed 1%. In this range the test is accurate to 0.2% of each solvent. L.T.W.

### Determination of Hydrophile-Lipophile Character of Surface-active Agents and Oils by Titration with Water

H. L. Greenwald, G. L. Brown, and M. N. Fineman Anal. Chem., 28, 1693–1697 (Nov. 1956) A benzene or dioxan soln, of an oil or a surface active agent is titrated with water to a dense-cloud end-point.

L.T.W.

#### Test-tube Scheme for Qualitative Testing of the Organic Constituents of Detergents

A. Hintermaier

World Congress on Surface-active Agents, 1, 301-309 (1954)

The physical state, odour, colour, solubility in water, foaming characteristics, ultraviolet fluorescence, and gelatin liquefaction are first noted. Differential solubilities in solvents, e.g. ethanol, and saponification tests complete the scheme.

#### Laboratory Evaluation of Detergent Power

Merlo and Coletti

World Congress on Surface-active Agents, 1, 247-251 (1954)

The Launder-Ometer appears to have too violent agitation to enable clear distinctions to be made between the powers of different detergents. Methods are described in which the soiled fabric is only gently moved or remains static and which permit better differentiation between 8.R.C. detergents.

#### Dishwashing Test as a Measure of Cleansing Power A. von Segesser and H. Stüpel

World Congress on Surface-active Agents, 2, 657-664 (1954)

Essentially, glass plates carrying a standard soiling of characterised pork fat were hand-washed in sequence for 45 sec. each in 0.5 g, active per litre, previously foamed up for approx. 1 min. by hand. The throughput before disappearance of foam was noted. The test is independent of operator and reproducible to I plate, relates closely to domestic conditions, and shows good distinction between various proprietary products.

#### Laboratory Evaluation of Detergents for Wool Piece Scouring

E. A. Leidelmeyer

World Congress on Surface-active Agents, 2, 451-461 (1954)

A model dolly is favoured and the published methods of six workers are compared. The effect of the rollers depends on the pressure, bath temperature, cloth speed, and presence of surfactants. The soiling method and the influence of the original pH of the wool are considered.

#### Triarylmethane Compounds as Redox Indicators in the Schoenemann Reaction. I- Mechanism of the Schoenemann Reaction

G. A. Grant, R. Blanchfield, and D. M. Smith Canadian J. Chem., 35, 40-47 (Jan. 1957)

#### Quantitative Analysis of Fibre Mixtures

A. Darbon and J. Jacquemart Bull. Inst. Text. France, (63), 97-104 (Oct. 1956) A microscopical method is based on the number of fibres of each component present in the sample (3000-4000 fibres) and their dimensions and densities. The wt. % of the ith component is given by  $100X_t/\Sigma X_t$ , where X= $Nd\varphi^2$  and N is the number of fibres of the ith component present, d its density, and  $\varphi$  the mean fibre diameter. If the departure from circular cross-section is marked, a correction factor is applied to q. As an example, the method is applied to the analysis of a ternary mixture of Fibravyl and two viscose rayons.

#### Maturity of Raw Cotton by the Triple Differential Dyeing Test and Use of the Micronaire Value

Bull, Inst. Text. France, (63), 15-44 (Oct. 1956) As a result of the study of the rates of sorption and descrption of the green and red dyes used in the differential dyeing test for the maturity of cotton, the author proposes a test requiring three different dyeings on successive samples, the dyebaths containing an increasing proportion of the green dye. The proportion of fibres dyed green in each test, together with the Micronaire value of the sample, allows the construction of the cumulative frequency curve of fibre fineness against weight per cent. The method gives a better indication of the distribution of fibre fineness than existing methods of determining maturity.

#### Chemical Damage in Wool. I-Determination by Paper Chromatography

W. H. Houff and R. H. Beaumont

Text. Research J., 26, 871-874 (Nov. 1956) An acid hydrolysate of the damaged wool sample is An acid hydrolysace of the damages wool sample is run on a two-dimensional paper chromatogram in the following solvents—(a) ethanol, butanol, water, propionic acid (10:10:5:2); (b) butanol, acetone, water, dicyclo-hexylamine (10:10:5:2). The amino-acid spots are made visible by spraying with ninhydrin. The solvent system used permits the detection of cysteic acid, produced by oxidative damage, and lanthionine, produced by alkali damage, as well as unchanged cystine. Visual estimations of the degree of damage by this method are compared with the reduction in force required to stretch the wet fibre after (a) chlorine treatments and (b) alkaline treatments of increasing severity.

#### Rapid Procedure for Estimation of Amino Acids by Direct Photometry on Filter-paper Chromatograms L. B. Rockland and J. C. Underwood

Anal. Chem., 28, 1679-1684 (Nov. 1956) Amino acids are estimated on paper chromatograms by photometric determination of total spot density after staining with ninhydrin. Photometry is facilitated by the use of a device for mounting the paper strips in front of the photocell. Complete amino-acid determinations may be made within 24 hr. The sensitivity is  $0.1-1.0 \mu g$ . for soln, containing as little as 150 µg./ml. L.T.W.

#### Concordance of Practical and Laboratory Results in Washing

K. J. Nieuwenhuis and K. H. Tan

World Congress on Surface-active Agents, 2,

522-538 (1954)

The Delft Experimental Bleaching Centre Machine operates differently from the majority of standard laboratory washing machines, having an automatically reversing internal perforated cylindrical cage inside an external cylindrical cage. It is, therefore, a miniature of the normal laundry machine, against which it was compared on six artificial soils. Good agreement was obtained with practical laundering when the Delft E.B.C. Soil was used (5 g. lampblack + 40 g. powdered silica + 40 g. edible olive oil + 10 g. beef fat + 5 g. Vaseline). S.R.C.

#### Method for Control of Washing

World Congress on Surface-active Agents, 2,

616-621 (1954)

A graphical method for assessing the efficiency of various conditions depends on photometric measurements on standard naturally soiled patterns before and after washing. The sample is measured over a squared surface and fixed by co-ordinate references giving a population of about 3,000,  $S_i$  representing the pre- and  $B_1$  the postseries. By arrangement of the  $S_1$  values as abscissae and the arithmetical means of the corresponding  $B_1$  values as ordinates, a curve results the slope of which shows zero bleaching at 45°, increasing with increased efficiency

#### Laboratory Dyeing Machine

J. Cegarra Sanchez

Ingenieria Textil, 23, 504-512 (Nov.-Dec. 1956) Automatic control of temperature and agitation with constant volume of bath are combined with the possibility of varying these factors independently. The liquor is heated by immersion in an electrically heated glycerol bath. Agitation is effected by oscillation of the pattern carrier, and evaporation is prevented by a condenser.

The dyebath receptacles are of special form.

S.R.C.

#### Precise Measurement of Fading on a Time-Intensity

Basis J. S. Mudd

J.S.D.C., 73, 47-52 (Feb. 1957)

Difficulties with B.S. 1006 are discussed, particularly with reference to its use for testing a wider range of materials than was originally intended. It is necessary to recognise as fading changes in appearance, resulting from the action of light, other than simple destruction of colour. The precise, if arbitrary, point at which a colour may be said to have faded can be determined by means of a combined chromaticity and luminance tolerance derived from measurements in C.I.E. terms. From a consideration of their spectral energy distributions, the artificial light sources in common use for fading tests could not be expected to give results comparable with those obtained with natural daylight, but the light from a xenon are is shown to have a similar effect to daylight. A simple type of fading lamp is described using a 150-watt xenon lamp. Since the light falling on a pattern can be maintained at constant intensity for a considerable time and the intensity can be accurately measured, the fastness to light of such a pattern can be precisely expressed in terms of the amount of light (in kilo-ft.-candle-hr.) required to fade the pattern to a predetermined colour tolerance.

#### Colorimetric Method for Determination of Glucosamine and Galactosamine

S. Roseman and I. Daffner

Anal. Chem., 28, 1743–1746 (Nov. 1956)
Hexosamines are determined after N-acetylation by a colorimetric method employing Ehrlich's reagent (p-dimethylaminobenzaldehyde). N-Acetylglucosamine and N-acetylglactosamine may be determined in admixture by a differential method.

L.T.W.

#### Determination of the Degree of Polymerisation of Cellulose Nitrates by means of Viscometric Measurements. I—Experimental

W. Lang

Scensk Papperstidning, 59, 819–828 (15 Dec. 1956) The viscometric method has been examined critically in order to detect possible sources of error and to ensure good reproducibility of the limiting viscosity values. Dry, purified cellulose nitrate keeps for five weeks without degradation, and the limiting viscosity of solutions in butyl acetate remains constant for eight days. It is essential to shake the freshly prepared solutions vigorously several hours before use. For good reproducibility, a nitration method which ensures constant N contents should be used. The Harland method was fourfit to be suitable for the determination of the N content. 33 references. K.W.

#### Detection of Traces of Formaldehyde in Dried Hide and Skin Material

A. Küntzel and P. Falcoz

Leder, 7, 107–109 (1956); J. Amer. Leather Chem. Assocn., 51, 567 (Oct. 1956) Some Persian dried goatskins that did not soak properly were tested for HCHO. In some of these skins HCHO could be detected, but in others, not. The question therefore arose as to whether HCHO could be present in amounts too small to be detected analytically, yet sufficient to harden the skin. The following quantitative test was tried: 6 g. of dry hide-powder was treated for 2 hr. with 0.1% (on hide-powder basis) of HCHO dissolved in 75 ml. of water. The pH was adjusted to 8.5 with NaOH. The mixture was then hydrolysed for 80 min, with 10 ml, of N-HCl in a pressure flask at 110°c. All the hide-powder dissolved. HCHO was determined by the method of Brochet and Cambier, in which it reacts with hydroxylamine hydrochloride, liberating an equivalent amount of acid, which is then titrated to pH 4-5 either with Bromophenol Blue or electrometrically. The results showed that there was a loss of about 20% of the HCHO. A series of dilutions of HCHO were tested qualitatively with the Gross-Bohle reagent—an SO<sub>2</sub>-decolorised solution of parafuchsine (rosaniline) which reddens on addition of traces of aldehyde. The greatest dilution that gave a positive test was 1:200,000. The chromotropic acid test, in which a violet-pink colour develops in the presence of HCHO in strong H<sub>2</sub>SO<sub>4</sub>, had a limit of 1:1,000,000, but MCHO in strong H<sub>2</sub>SO<sub>4</sub>, and a limit of 1:1,000,000, but when hide-powder was treated with formaldehyde and hydrolysed as above, the limit was reduced to 1:800,000. Drops (0.05 ml.) of HCHO of different dilutions were placed on strips of dry, split collagen, dried, then soaked the following day. Hard spots that did not soak back well and appeared similar to those on the Persian skins were found at dilutions from 1:1 to 1:1000, but no hard spot was found at the spot treated with the 1:10,000 dilution. The hard spots were cut out, hydrolysed (considerable dilution was necessary for hydrolysis), and tested by the chromotropic acid test. Spots from the 1:100 dilution gave positive, but those from the 1:1000 dilution negative, tests for HCHO. It is therefore possible for skins to have been treated with HCHO and yet to give no positive test for it.

C.J.W.H.

#### Thiohydantoin Method for the Determination of the Terminal Carboxyl Amino Acids of Collagen

C. Deasy
J. Amer. Leather Chem. Assocn., 51, 584-591 (Nov. 1956)
A slight modification of the method of Baptist and Bull
(J. Amer. Chem. Soc., 75, 1727 (1953)) has been applied to
collagen. The amino acids were identified by paper
chromatography. Glycine, alanine, and leucine (or isoleucine) have been found, and they may be regarded as the
terminal carboxyl amino acids of collagen. The method
has some limitations— (i) it fails to detect lysine, arginine,
glutamic acid, and aspartic acid when they occur as Cterminal acids, and these are thus not definitely excluded
as possible C-terminal acids of collagen or gelatin; (ii) since
the last step in the method is an alkaline hydrolysis, the
glycine and alanine found in the final soln, may have been
formed from serine and threonine. Independent evidence
in support of the findings of the investigation is discussed.

#### PATENT

#### Measuring Thickness

BP 759,196

A strip of flexible sheet material, e.g. a plastic film, is advanced intermittently between the movable foot of a stationary thickness gauge and a stationary anvil towards which the foot is urged. The distance between the gauge and the anvil is fixed at the nominal sheet thickness, and any variations of the thickness are recorded by the displacement of the foot from the anvil on a chart. Point-to-point variations of a wider strip can be recorded by mounting several stationary thickness gauges side by side.

#### XV-MISCELLANEOUS

#### Recent Advances in Surface Chemistry

J. J. Kipling

J. Oil & Col. Chem. Assocn., 39, 641–652 (Sept. 1956) Recent work on physical adsorption by solids from binary liquid mixtures is reviewed, with particular reference to methods for determining the adsorption of each component separately. The results are consistent with a monolayer theory of adsorption. The orientation of adsorbed molecules is being given greater attention in terms of their precise shape and size; this is illustrated by reference to films of stearic acid adsorbed on water. Greater attention should be paid to this point in the determination of the surface areas of solids. J.W.D.

#### Crystal Symmetry, and the Adsorption of Dyes by Growing Crystals. I— Ammonium Nitrate IV

Ammonium nitrate IV (I), when crystallised from saturated solutions containing a dissolved dye, frequently gives platy habit-modified cryst. with pleochroic coloured regions containing adsorbed dye. The suitability for dye adsorption of the principal low-index planes of I is discussed in the light of the scheme proposed previously (cf. Trans. Faruday Soc., 51, 973, 1142 (1955)) to explain modifications of cryst. habit by dyes. It is shown how the observed pleochroic inclusions are essentially related to the cryst. symmetry of I and how the habit modifications are produced.

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All inquiries relating to Advertisements should be addressed to The General Secretary, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire.

Replies may be addressed Box —, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1,

YORKSHIRE, where all communications relating to these Advertisements are treated in strict confidence.

#### APPOINTMENTS VACANT

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Approximate are invited from chemists of texture technicipus with a degree or equivalent qualification, and preferably with experience in the textile field. The work involves some customer contact and applicants must be prepared for a limited amount of travelling. An attractive and progressive salary is offered, and the post carries membership of the Company's non-contributory pension scheme. In addition, house-purchase facilities may be available.

available.

Applications, stating briefly, age, qualifications and experience, should be addressed to the Staff Officer (Ref. 360), Albright & Wilson, Ltd., (with whom Midland Silicones Ltd., is associated), P.O. Box. 3, Oldbury, Birmingham.

A SSISTANT required to Director of Patent Licensing Division of British Company in Midlands. Age 35-45 years. University Degree: at least one foreign language: experience of the wool textile dyeing and unishing trade essential, although duties will be commercial rather than technical. Considerable weight will be given to the qualities of self-reliance, judgment and initiative: the position will be exacting and therefore carries corresponding remuneration.

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Applications, which will be treated in confidence, should be made in writing to—

The Personnel Manager, BX Plantics 144

The Personnel Manager, BX Plastics Ltd., Brantham Works, near Manningtree, Essex

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VACANCIES for two graduates exist in the laboratories of the Dyers and Cleaners' Research Organisation. Candidates aged 25 to 35 should preferably have some textile experience or qualification although this is not essential. Salary according to age and experience, Apply to Director of Research, 4 Lifton Flace, Leeda 21.73. Telephone, Leeda 21.73.

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DEPARTMENT OF TEXTILE INDUSTRIES

A PPLICATIONS are invited for a Fellowship which has been established by Bleachers' Association Ltd. for research on celluloside fibres. The value of the Fellowship, which is tenable for one year in the first instance, with possibility of renewal, is £600 per annum together with superannuation contributions under the F.8.8.U, scheme. Candidates should have leaning towards physical or physical-organic chemistry. Applications, stating date of birth, qualifications and experience, together with the names of two referees should be sent to the Registrar, The University, Leeda 2, from whom further inquiries may be made, not later than 11th May 1957.

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DEPARTMENT OF TEXTILE INDUSTRIES

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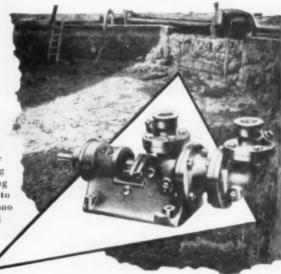
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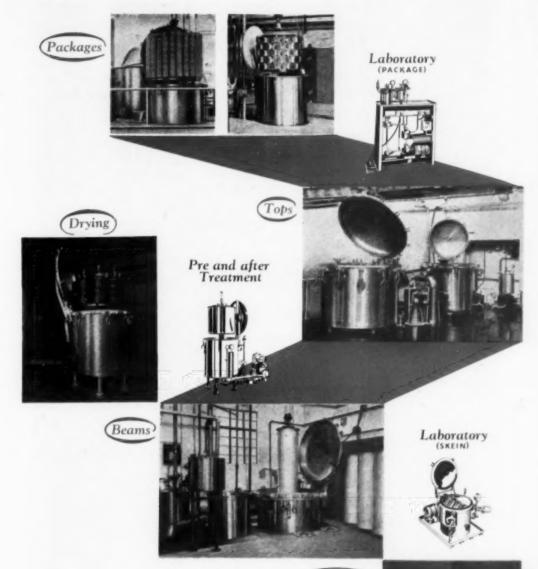
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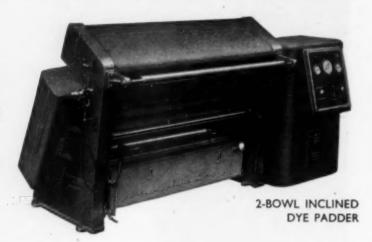
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